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NBS REPORT

6785

**THIRD PROGRESS REPORT**

to

**National Aeronautics and Space Administration**

on

**Cryogenic Research and Development**

for

**Period Ending June 30, 1961**

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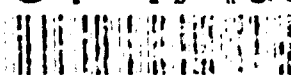
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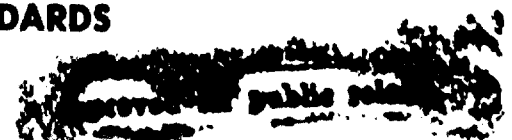
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**U. S. DEPARTMENT OF COMMERCE  
NATIONAL BUREAU OF STANDARDS**

**BOULDER LABORATORIES  
Boulder, Colorado**



*SN-91671  
PR#3  
June 30, 1961*

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# NATIONAL BUREAU OF STANDARDS REPORT

NBS PROJECT

81410, 81420  
81430, 81450

June 30, 1961

NBS REPORT

6785

## THIRD PROGRESS REPORT

to

National Aeronautics and Space Administration

on

Cryogenic Research and Development

for

Period Ending June 30, 1961

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U. S. DEPARTMENT OF COMMERCE  
NATIONAL BUREAU OF STANDARDS  
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## 1. Physical Properties of Fluid Hydrogen

### 1.1 Heat Capacity and PVT Measurements

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H. M. Roder, Dr. L. A. Weber, Dr. B. A. Younglove

#### 1.1.1 Summary

Overall objective of this project is the preparation of a thermodynamic network for para hydrogen from 17° to 100°K and from 2 to 350 atm. Now in preparation are the detailed physical properties data necessary for the thermodynamic computations. The PVT experimental work is completed. Specific heats of saturated liquid are nearly completed. Specific heats of compressed liquid and fluid at constant densities near the liquid triple-point density will be determined shortly. These data will establish a firm relationship between values of the thermodynamic properties for compressed liquid and for the vapor, gaseous and fluid states. In course of preparation, by computational methods, are the densities of saturated vapor; the heats of vaporization; the pressure-density relation for saturated liquid; a smoothed, interpolated and T'-scale-adjusted vapor-pressure relationship; values of thermodynamic properties for saturated liquid relative to spectroscopically-determined, ideal gaseous states, and also relative to perfect crystal at  $T = 0$ . A manuscript describing the entire apparatus installation and calibration has been submitted to the NBS Journal of Research. A manuscript on saturated liquid densities has been submitted to the journal, *Cryogenics*.

#### 1.1.2 Description of Data Tables

In this report the substance is para hydrogen and the units are cubic centimeters, atmospheres, gram-moles and degrees

Kelvin on the NBS 1955 temperature scale unless noted otherwise. Value of the gas constant used is  $R = 82.057$ . Following tables include all adjustments required for a permanent pipet volume increased by factor 1.00263, which deformation probably occurred during Series I experiments.

a. Tables I-a and I-b, Virial Coefficients. Virial coefficients from Series III experiments for

$$Z \equiv Pv/RT = 1 + B/v + C/v^2 + \dots \quad (1)$$

in the form

$$\phi \equiv RT(Z-1)v = RTB + RTC/v + \dots \quad (2)$$

are obtained by least squares from the linear-dependence of  $\phi$  upon density, as given under Exp. by tables I-a and I-b, valid for densities below nine gram-moles per liter. Bracketed entries in these tables are extrapolated. Under heading RP 1932 are values for normal hydrogen from the compendium of Woolley, Scott and Brickwedde, J. Research NBS 41, 379 (1948).

b. Tables II and III, Compressed Liquid and Fluid Regions.

Pressures are represented along isotherms by polynomials in the adjusted densities,  $\sigma \equiv 1/v$ ,

$$P = A_1 + A_2 \sigma + A_3 \sigma^2 + \dots \quad (3)$$

The coefficients, determined by least squares from Series II data, are presented by table II. Temperature for each isotherm appears above and to the left of each double row of coefficients. This isotherm representation permits computation of table III, giving pressures at uniform densities and temperatures. Densities for table III are in g.mol./1000 cm<sup>3</sup>. Entries above and below the dividing lines, near top and bottom of each column, are extrapolated beyond observed pressures.



Table I-a. Second Virial Coefficient

Units of  $\text{cm}^3$ , atm., g. mol., deg. K

<u>T, °K</u>	<u>-RTB · 10<sup>-5</sup></u>		<u>d(RTB)/dT · 10<sup>-3</sup></u>	
	<u>Exp.</u>	<u>RP 1932</u>	<u>Exp.</u>	<u>RP 1932</u>
14	(2.83)	2.830	(8.20)	8.20
15	(2.75)		(7.46)	
16	(2.68)	2.681	(6.86)	6.86
17	(2.61)		(6.35)	
18	(2.55)	2.554	(5.95)	5.95
19	(2.49)		(5.60)	
20	(2.44)	2.442	(5.31)	5.31
21	(2.39)		(5.05)	
22	(2.33)	2.341	(4.82)	4.87
23	(2.28)		(4.61)	
24	2.229	2.247	4.41	4.53
25	2.185		4.24	
26	2.139	2.159	4.09	4.27
27	2.098		3.97	
28	2.059	2.075	3.85	4.04
29	2.023		3.75	
30	1.987	1.996	3.66	3.84
31	1.952		3.58	
32	1.917	1.921	3.50	3.66
33	1.883		3.44	
34	1.848	1.849	3.37	3.49
35	1.814		3.31	
36	1.780	1.781	3.25	3.33
37	1.747		3.20	
38	1.714	1.715	3.15	3.20
39	1.683		3.10	
40	1.651	1.653	3.05	3.10
42	1.591	1.591	2.96	3.02
44	1.533	1.532	2.88	2.95
46	1.478	1.473	2.80	2.89
48	1.423	1.416	2.73	2.83
50	1.369	1.360	2.67	2.77
55	1.239		2.55	
56		1.198		2.67

Table I-a. Second Virial Coefficient (Continued)

Units of  $\text{cm}^3$ , atm., g. mol., deg. K

<u>T, °K</u>	<u>-RTB · 10<sup>-5</sup></u>		<u>d(RTB)/dT · 10<sup>-3</sup></u>	
	<u>Exp.</u>	<u>RP 1932</u>	<u>Exp.</u>	<u>RP 1932</u>
60	1.116		2.47	
65	0.992		2.40	
70	0.873		2.35	
75	0.762		2.31	
80	0.647		2.28	
85	0.531		2.24	
90	0.417		2.21	
95	0.310		2.19	
100	0.205		2.16	

Table I-b. Third Virial Coefficient

Units of  $\text{cm}^3$ , atm., g. mol., deg. K

T, °K	RTC. $10^{-6}$			$\Delta(\text{RTC})/\Delta T. 10^{-4}$	
	Exp.	Smoothed	RP 1932	Smoothed	RP 1932
14		(1.83)		( 9.3)	
15		(1.93)		( 9.9)	
16		(2.03)		(10.6)	
17		(2.14)		(11.2)	
18		(2.25)		(11.9)	
19		(2.37)		(12.5)	
20		(2.50)		(13.1)	
21		(2.63)		(13.7)	
22		(2.77)		(14.2)	
23		(2.915)		(14.7)	
24	3.053	3.060		15.0	
25	3.432	3.215		15.2	
26	3.273	3.370		15.2	
27	3.519	3.525		14.9	
28	3.647	3.685		14.4	
29	3.826	3.825		13.2	
30	3.938	3.945		11.1	
31	4.021	4.040		7.9	
32	4.096	4.100		3.9	
33	4.124	4.120		0.6	
34	4.110	4.115	4.171	-1.4	-4.4
35	4.103	4.095		-2.4	
36	4.073	4.065	4.089	-2.8	-3.7
37	4.043	4.040		-2.7	
38	4.020	4.020	4.020	-2.5	-3.2
39	3.998	4.000		-2.2	
40	3.966	3.980	3.962	-1.9	-2.6
42	3.934	3.950	3.914	-1.2	-2.1
44	3.920	3.935	3.876	-0.6	-1.7
46	3.922	3.925	3.846	-0.1	-1.3
48	3.929	3.920	3.825	+0.4	-0.9
50	3.926	3.935	3.811	0.7	-0.5
55	3.971	3.990		1.3	
56			3.806		+0.3

Table I-b. Third Virial Coefficient (Continued)

Units of  $\text{cm}^3$ , atm., g. mol., deg. K

T, °K	RTC · 10 <sup>-6</sup>			$\Delta(\text{RTC})/\Delta T \cdot 10^{-4}$	
	Exp.	Smoothed	RP 1932	Smoothed	RP 1932
60	4.074	4.065		1.7	
65	4.143	4.150		2.0	
70	4.232	4.250		2.2	
75	4.414	4.360		2.4	
80	4.514	4.470		2.5	
85	4.587	4.590		2.6	
90	4.688	4.720		2.7	
95	4.849	4.850		2.8	
100	5.007	4.990		2.8	

Table II(a)

POLYNOMIAL CONSTANTS FOR REVISED ISOTHERMS

17.							
A(1)=	-.2438491533E+4	A(2)=	.2183803894E+6	A(3)=	-.6991161219E+7		
A(4)=	.7764605231E+8						
18.							
A(1)=	.7013777525E+5	A(2)=	-.7189900233E+7	A(3)=	.2765239294E+9		
A(4)=	-.4743120165E+10	A(5)=	.3073033490E+11				
19.							
A(1)=	-.6989362290E+5	A(2)=	.9049673649E+7	A(3)=	-.4672897569E+9		
A(4)=	.1202211660E+11	A(5)=	-.1543429920E+12	A(6)=	.7946580691E+12		
20.							
A(1)=	-.2254318359E+6	A(2)=	.2911965136E+8	A(3)=	-.1502470236E+10		
A(4)=	.3870063625E+11	A(5)=	-.4978787887E+12	A(6)=	.2562864383E+13		
21.							
A(1)=	-.1342809016E+5	A(2)=	.1694631524E+7	A(3)=	-.8480700146E+8		
A(4)=	.2096870700E+10	A(5)=	-.2580137441E+11	A(6)=	.1299418465E+12		
22.							
A(1)=	-.3583802425E+5	A(2)=	.4618805983E+7	A(3)=	-.2372094855E+9		
A(4)=	.6063035984E+10	A(5)=	-.7733189085E+11	A(6)=	.3973311238E+12		
23.							
A(1)=	-.2714158013E+4	A(2)=	.3075711483E+6	A(3)=	-.1313194972E+8		
A(4)=	.2500407516E+9	A(5)=	-.2056246500E+10	A(6)=	.8050883349E+10		
24.							
A(1)=	.1654487568E+5	A(2)=	-.2184788079E+7	A(3)=	.1156131935E+9		
A(4)=	-.3067675537E+10	A(5)=	.4060413169E+11	A(6)=	-.2109275905E+12		
25.							
A(1)=	.2071615707E+4	A(2)=	-.2877690160E+6	A(3)=	.1639927601E+8		
A(4)=	-.4790607599E+9	A(5)=	.6918127881E+10	A(6)=	-.3602601593E+11		
26.							
A(1)=	-.1077533592E+3	A(2)=	.1040854433E+5	A(3)=	.1140264456E+6		
A(4)=	-.3478581672E+8	A(5)=	.8742378097E+9	A(6)=	-.3235709802E+10		
27.							
A(1)=	.5135780451E+3	A(2)=	-.7543052590E+5	A(3)=	.4835797214E+7		
A(4)=	-.1635906656E+9	A(5)=	.2626725190E+10	A(6)=	-.1275841466E+11		
28.							
A(1)=	-.2435909244E+2	A(2)=	-.5362369801E+4	A(3)=	.1198281126E+7		
A(4)=	-.6901361784E+8	A(5)=	.1403732922E+10	A(6)=	-.6474043949E+10		
29.							
A(1)=	-.8368161770E+3	A(2)=	.1086448254E+6	A(3)=	-.5177169738E+7		
A(4)=	.1090481884E+9	A(5)=	-.1069637056E+10	A(6)=	.7189124536E+10		

Table II(b)

30.							
A(1)=	.5606041669E+3	A(2)=	-.9153081757E+5	A(3)=	.6229597109E+7		
A(4)=	-.2137171922E+9	A(5)=	.3476301198E+10	A(6)=	-.1831366371E+11		
31.							
A(1)=	.2553396629E+3	A(2)=	-.4652766080E+5	A(3)=	.3587773320E+7		
A(4)=	-.1360748682E+9	A(5)=	.2345330674E+10	A(6)=	-.1178470095E+11		
32.							
A(1)=	.1379922212E+3	A(2)=	-.2817973335E+5	A(3)=	.2444248723E+7		
A(4)=	-.1001056829E+9	A(5)=	.1787235536E+10	A(6)=	-.8365794837E+10		
33.							
A(1)=	.1390453106E+3	A(2)=	-.2793285192E+5	A(3)=	.2404675917E+7		
A(4)=	-.9770055517E+8	A(5)=	.1737689386E+10	A(6)=	-.8017689700E+10		
34.							
A(1)=	.1308040484E+3	A(2)=	-.2614178026E+5	A(3)=	.2266579485E+7		
A(4)=	-.9224669537E+8	A(5)=	.1642029160E+10	A(6)=	-.7395129824E+10		
35.							
A(1)=	.1303151614E+3	A(2)=	-.2565492386E+5	A(3)=	.2216095655E+7		
A(4)=	-.8969970762E+8	A(5)=	.1593742590E+10	A(6)=	-.7075411354E+10		
36.							
A(1)=	.1240366301E+3	A(2)=	-.2407339486E+5	A(3)=	.2084976854E+7		
A(4)=	-.8423031310E+8	A(5)=	.1493104616E+10	A(6)=	-.6384555723E+10		
37.							
A(1)=	.1225028239E+3	A(2)=	-.2349903433E+5	A(3)=	.2036851098E+7		
A(4)=	-.8205996730E+8	A(5)=	.1455753505E+10	A(6)=	-.6163977191E+10		
38.							
A(1)=	.1119747027E+3	A(2)=	-.2115631264E+5	A(3)=	.1853119336E+7		
A(4)=	-.7480256544E+8	A(5)=	.1324900231E+10	A(6)=	-.5269386480E+10		
39.							
A(1)=	.1124295109E+3	A(2)=	-.2102706712E+5	A(3)=	.1843152149E+7		
A(4)=	-.7417780518E+8	A(5)=	.1317166865E+10	A(6)=	-.5265272390E+10		
40.							
A(1)=	.1053917976E+3	A(2)=	-.1940803885E+5	A(3)=	.1717349801E+7		
A(4)=	-.6913829430E+8	A(5)=	.1226860836E+10	A(6)=	-.4654736080E+10		
42.							
A(1)=	.9955734284E+2	A(2)=	-.1791259323E+5	A(3)=	.1609172437E+7		
A(4)=	-.6477751878E+8	A(5)=	.1156699461E+10	A(6)=	-.4263542234E+10		
44.							
A(1)=	.9666801440E+2	A(2)=	-.1702714325E+5	A(3)=	.1550122819E+7		
A(4)=	-.6232099793E+8	A(5)=	.1122000082E+10	A(6)=	-.4128067718E+10		

Table II(c)

46.							
A(1)=	.8872775941E+2	A(2)=	-.1525645588E+5	A(3)=	.1430073442E+7		
A(4)=	-.5776982031E+8	A(5)=	.1051007661E+10	A(6)=	-.3738059166E+10		
48.							
A(1)=	.7220497817E+2	A(2)=	-.1162520350E+5	A(3)=	.1153143512E+7		
A(4)=	-.4676101284E+8	A(5)=	.8500157041E+9	A(6)=	-.2324793971E+10		
50.							
A(1)=	.7500637784E+2	A(2)=	-.1204805268E+5	A(3)=	.1209092770E+7		
A(4)=	-.4911436839E+8	A(5)=	.9112281682E+9	A(6)=	-.2926320117E+10		
55.							
A(1)=	.5547510654E+2	A(2)=	-.7466254043E+4	A(3)=	.8768165023E+6		
A(4)=	-.3550310684E+8	A(5)=	.6673341977E+9	A(6)=	-.1251686832E+10		
60.							
A(1)=	.3487437747E+2	A(2)=	-.2696580534E+4	A(3)=	.5302788190E+6		
A(4)=	-.2126585564E+8	A(5)=	.4074952364E+9	A(6)=	.5859404725E+9		
65.							
A(1)=	.1574950988E+2	A(2)=	.1876167588E+4	A(3)=	.1888965823E+6		
A(4)=	-.6693380078E+7	A(5)=	.1278416144E+9	A(6)=	.2691617494E+10		
70.							
A(1)=	.3013700679E+2	A(2)=	-.9743211592E+3	A(3)=	.4933306556E+6		
A(4)=	-.1970370357E+8	A(5)=	.4262154895E+9	A(6)=	.3130942589E+8		
75.							
A(1)=	.4268725563E+2	A(2)=	-.3335378475E+4	A(3)=	.7479339288E+6		
A(4)=	-.3028515725E+8	A(5)=	.6668995905E+9	A(6)=	-.2101337873E+10		
80.							
A(1)=	.7774744042E+2	A(2)=	-.1069315768E+5	A(3)=	.1440408319E+7		
A(4)=	-.5981691753E+8	A(5)=	.1312408340E+10	A(6)=	-.7657277523E+10		
85.							
A(1)=	.2710929772E+2	A(2)=	.1642370928E+4	A(3)=	.3397552134E+6		
A(4)=	-.8484502872E+7	A(5)=	.1514658698E+9	A(6)=	.2771419306E+10		
90.							
A(1)=	-.6430160122E+2	A(2)=	.2326195507E+5	A(3)=	-.1594851007E+7		
A(4)=	.7979725121E+8	A(5)=	-.1817785954E+10	A(6)=	.2018378418E+11		
95.							
A(1)=	.2834661374E+3	A(2)=	-.5903323152E+5	A(3)=	.6210235760E+7		
A(4)=	-.2839926589E+9	A(5)=	.6604958634E+10	A(6)=	-.5708083908E+11		
100.							
A(1)=	.3030719867E+3	A(2)=	-.6370804359E+5	A(3)=	.6744388597E+7		
A(4)=	-.3106286889E+9	A(5)=	.7287240620E+10	A(6)=	-.6395117418E+11		

Table III(a)

## PRESSURES AT UNIFORM DENSITIES AND TEMPERATURES

TEMPERATURE IN DEGREES KELVIN

DENSITY	17	18	19	20	21	22	23	24
33.0								<u>5.139</u>
33.5							<u>2.469</u>	<u>10.310</u>
34.0							<u>8.288</u>	<u>16.192</u>
34.5						6.668	14.744	22.793
35.0					5.370	13.640	21.884	30.129
35.5				<u>4.389</u>	12.826	21.299	29.754	38.222
36.0			<u>3.951</u>	<u>12.501</u>	21.055	29.717	38.399	47.098
36.5		<u>4.091</u>	<u>12.532</u>	21.321	30.109	38.957	47.865	56.791
37.0	3.689	<u>13.055</u>	22.016	30.988	40.035	49.072	58.193	67.336
37.5	<u>14.069</u>	23.252	32.454	41.599	50.877	60.108	69.426	78.772
38.0	<u>25.321</u>	34.558	43.886	53.211	62.676	72.105	81.606	91.142
38.5	37.503	46.896	56.341	65.860	75.473	85.101	94.773	104.487
39.0	50.674	60.234	69.846	79.563	89.305	99.129	108.968	118.853
39.5	64.891	74.589	84.424	94.330	104.208	114.220	124.230	134.283
40.0	80.213	90.020	100.101	110.175	120.220	130.408	140.596	150.820
40.5	96.699	106.635	116.905	127.122	137.375	147.726	158.105	168.505
41.0		124.588	134.871	145.221	155.710	166.212	176.794	187.379
41.5			154.045	164.551	175.261	185.909	196.699	207.478
42.0			174.485	185.232	196.066	206.863	217.856	228.732
42.5				207.436	218.166	229.132	240.300	251.770
43.0					<u>241.602</u>	252.780	264.067	275.413
43.5					<u>266.420</u>	<u>277.884</u>	289.189	300.678
44.0						<u>304.530</u>	<u>315.701</u>	<u>327.272</u>
44.5							<u>343.636</u>	<u>355.195</u>



Table III(b)

TEMPERATURE IN DEGREES KELVIN								
DENSITY	25	26	27	28	29	30	31	32
22.5								10.890
23.0								<u>11.177</u>
23.5								11.559
24.0								12.053
24.5								12.676
25.0							9.396	13.445
25.5							<u>10.127</u>	14.380
26.0						6.712	11.045	15.500
26.5						<u>7.597</u>	12.170	16.829
27.0						8.721	13.525	18.387
27.5					4.871	10.105	15.133	20.198
28.0					<u>6.432</u>	11.770	17.016	22.287
28.5				2.895	8.258	13.740	19.202	24.680
29.0				<u>4.757</u>	10.384	16.041	21.715	27.401
29.5			1.240	6.969	12.842	18.695	24.583	30.480
30.0			3.550	9.560	15.667	21.735	27.834	33.943
30.5			6.286	12.560	18.893	25.185	31.497	37.819
31.0		2.928	<u>9.477</u>	16.001	22.554	29.075	35.602	42.139
31.5		6.386	13.151	19.915	26.685	33.437	40.180	46.933
32.0	<u>3.353</u>	<u>10.354</u>	17.338	24.335	31.319	38.302	45.262	52.231
32.5	7.637	14.865	22.070	29.292	36.490	43.702	50.880	58.067
33.0	12.504	19.952	27.379	34.821	42.234	49.670	57.067	64.471
33.5	17.986	25.649	33.297	40.955	49.534	56.240	63.858	71.479
34.0	24.114	31.990	39.858	47.730	55.576	63.447	71.285	79.123
34.5	30.922	39.010	47.095	55.180	63.244	71.327	79.385	87.438
35.0	38.445	46.744	55.045	63.341	71.623	79.914	88.191	96.458
35.5	46.718	55.228	63.742	72.249	80.749	89.245	97.740	106.220
36.0	55.777	64.498	73.223	81.940	90.657	99.357	108.068	116.759
36.5	65.660	74.591	83.525	92.451	101.384	110.287	119.212	128.112
37.0	76.405	85.544	94.684	103.819	112.965	122.072	131.207	140.315
37.5	88.051	97.395	106.738	116.081	125.438	134.749	144.092	153.406
38.0	100.636	110.181	119.726	129.276	138.839	148.356	157.904	167.422
38.5	114.199	123.940	133.686	143.440	153.206	162.931	172.680	182.402
39.0	128.780	138.712	148.656	158.613	168.577	178.511	188.458	198.383
39.5	144.418	154.536	164.676	174.833	184.990	195.135	205.276	215.403
40.0	161.151	171.451	181.785	192.139	202.485	212.838	223.172	233.503
40.5	179.020	189.496	200.021	210.569	221.100	231.660	242.184	252.719
41.0	198.061	208.712	219.425	230.162	240.876	251.636	262.350	273.093
41.5	218.313	229.140	240.037	250.959	261.852	272.803	283.707	294.662
42.0	239.813	250.820	261.894	272.998	284.071	295.197	306.294	<u>317.466</u>
42.5	262.597	273.793	285.038	296.318	307.575	318.854	330.148	341.545
43.0	286.700	298.101	309.507	<u>320.959</u>	<u>332.405</u>	<u>343.810</u>	<u>355.307</u>	366.937
43.5	312.156	<u>323.786</u>	<u>335.342</u>	346.962	358.604	370.098	381.807	393.683
44.0	<u>338.999</u>	350.889	362.580	374.364	386.217	397.753		
44.5	367.259	379.453	391.261	403.206				

Table III(c)

## TEMPERATURE IN DEGREES KELVIN

DENSITY	33	34	35	36	37	38	39	40
16.5		14.820	16.874	18.948	21.040	23.142	25.254	27.372
17.0		14.871	17.003	19.164	21.343	23.539	25.740	27.952
17.5		14.945	17.162	19.412	21.682	23.974	26.268	28.578
18.0	12.845	15.041	17.348	19.693	22.058	24.449	26.840	29.249
18.5	12.855	15.160	17.564	20.007	22.472	24.965	27.458	29.970
19.0	12.886	15.305	17.811	20.357	22.928	25.525	28.125	30.743
19.5	12.942	15.479	18.093	20.747	23.428	26.134	28.845	31.573
20.0	13.028	15.687	18.415	21.183	23.979	26.798	29.624	32.467
20.5	13.150	15.936	18.784	21.671	24.586	27.523	30.470	33.432
21.0	13.315	16.233	19.207	22.219	25.259	28.318	31.391	34.475
21.5	13.533	16.587	19.694	22.835	26.006	29.193	32.395	35.607
22.0	13.814	17.010	20.254	23.531	26.838	30.157	33.493	36.838
22.5	14.170	17.512	20.900	24.319	27.765	31.223	34.698	38.180
23.0	14.613	18.108	21.645	25.210	28.802	32.403	36.021	39.646
23.5	15.158	18.810	22.502	26.219	29.961	33.710	37.477	41.249
24.0	15.820	19.634	23.486	27.360	31.258	35.161	39.081	43.004
24.5	16.615	20.597	24.614	28.651	32.709	36.771	40.847	44.927
25.0	17.562	21.717	25.904	30.108	34.331	38.557	42.794	47.035
25.5	18.680	23.011	27.373	31.750	36.142	40.536	44.940	49.345
26.0	19.988	24.501	29.042	33.596	38.161	42.729	47.302	51.878
26.5	21.508	26.207	30.931	35.667	40.409	45.155	49.902	54.652
27.0	23.261	28.150	33.062	37.983	42.908	47.836	52.760	57.687
27.5	25.271	30.354	35.458	40.568	45.678	50.792	55.898	61.007
28.0	27.563	32.843	38.141	43.445	48.744	54.047	59.339	64.633
28.5	30.160	35.641	41.137	46.637	52.129	57.625	63.106	68.588
29.0	33.089	38.775	44.472	50.171	55.859	61.551	67.223	72.897
29.5	36.377	42.269	48.170	54.071	59.958	65.849	71.717	77.586
30.0	40.052	46.153	52.260	58.366	64.454	70.547	76.613	82.679
30.5	44.142	50.455	56.770	63.082	69.375	75.670	81.938	88.203
31.0	48.676	55.203	61.728	68.248	74.748	81.249	87.720	94.187
31.5	53.686	60.427	67.164	73.894	80.602	87.310	93.987	100.659
32.0	59.201	66.158	73.108	80.049	86.968	93.884	100.769	107.647
32.5	65.253	72.427	79.592	86.744	93.875	101.000	108.096	115.181
33.0	71.875	79.266	86.646	94.012	101.355	108.691	115.999	123.293
33.5	79.100	86.709	94.304	101.883	109.441	116.987	124.508	132.012
34.0	86.961	94.787	102.598	110.391	118.163	125.921	133.656	141.372
34.5	95.492	103.536	111.563	119.569	127.557	135.526	143.476	151.404
35.0	104.728	112.988	121.231	129.451	137.655	145.835	154.002	162.142
35.5	114.705	123.181	131.639	140.072	148.491	156.884	165.266	173.620
36.0	125.457	134.148	142.821	151.468	160.102	168.706	177.304	185.872
36.5	137.022	145.927	154.813	163.673	172.521	181.337	190.151	198.934
37.0	149.436	158.553	167.652	176.724	185.786	194.813	203.843	212.841
37.5	162.736	172.064	181.374	190.659	199.933	209.172	218.415	227.629
38.0	176.960	186.496	196.016	205.513	214.998	224.449	233.905	243.336
38.5	192.145	201.888	211.617	221.326	231.020	240.683	250.350	259.998
39.0	208.329	218.278	228.214	238.134	248.036	257.912	267.788	277.653
39.5	225.552	235.704	245.845	255.977	266.084	276.174	286.256	296.340
40.0	243.851	254.205	264.549	274.893	285.203	295.508	305.794	316.098
40.5	263.265	273.819	284.365	294.922	305.432	315.954	326.440	336.965
41.0	283.834	294.587	305.332	316.103	326.811	337.552	348.234	358.982
41.5	305.598	316.547	327.490	338.475	349.378	360.342	371.216	382.189
42.0	328.594	339.739	350.877	362.080	373.174	384.364	395.426	
42.5	352.864	364.203	375.535	386.957	398.240			
43.0	378.447	389.979	401.503					

Table III(d)

TEMPERATURE IN DEGREES KELVIN								
DENSITY	42	44	46	48	50	55	60	65
16.5	31.628	35.902	40.154	44.436	48.723	59.443	70.140	80.833
17.0	32.397	36.858	41.310	45.791	50.270	61.481	72.670	83.850
17.5	33.217	37.872	42.527	47.211	51.888	63.600	75.291	86.968
18.0	34.089	38.946	43.809	48.698	53.580	65.806	78.008	90.194
18.5	35.018	40.082	45.159	50.258	55.351	68.103	80.830	93.536
19.0	36.006	41.286	46.581	51.896	57.207	70.500	83.764	97.004
19.5	37.059	42.561	48.082	53.618	59.154	73.002	86.818	100.606
20.0	38.183	43.916	49.669	55.433	61.200	75.620	90.002	104.352
20.5	39.386	45.358	51.349	57.349	63.353	78.362	93.325	108.254
21.0	40.677	46.896	53.133	59.375	65.624	81.237	96.799	112.321
21.5	42.064	48.538	55.029	61.522	68.023	84.258	100.435	116.565
22.0	43.560	50.297	57.048	63.801	70.560	87.436	104.244	120.999
22.5	45.175	52.184	59.204	66.223	73.248	90.783	108.239	125.636
23.0	46.922	54.211	61.508	68.803	76.101	94.312	112.434	130.489
23.5	48.816	56.393	63.975	71.554	79.132	98.038	116.843	135.572
24.0	50.870	58.744	66.619	74.490	82.356	101.975	121.481	140.900
24.5	53.101	61.281	69.457	77.628	85.789	106.139	126.363	146.488
25.0	55.526	64.019	72.504	80.983	89.447	110.547	131.505	152.353
25.5	58.162	66.976	75.779	84.574	93.348	115.215	136.925	158.512
26.0	61.028	70.171	79.299	88.418	97.510	120.162	142.640	164.982
26.5	64.143	73.624	83.085	92.535	101.952	125.406	148.669	171.781
27.0	67.528	77.354	87.155	96.944	106.694	130.966	155.031	178.928
27.5	71.205	81.383	91.532	101.666	111.756	136.864	161.746	186.443
28.0	75.195	85.732	96.236	106.723	117.160	143.119	168.834	194.346
28.5	79.522	90.425	101.291	112.136	122.927	149.754	176.317	202.659
29.0	84.209	95.485	106.720	117.929	129.082	156.790	184.217	211.404
29.5	89.281	100.937	112.547	124.126	135.646	164.252	192.557	220.602
30.0	94.764	106.805	118.796	130.751	142.646	172.163	201.359	230.278
30.5	100.685	113.117	125.495	137.829	150.105	180.548	210.650	240.456
31.0	107.069	119.897	132.668	145.388	158.049	189.431	220.453	251.161
31.5	113.946	127.175	140.343	153.453	166.505	198.840	230.794	262.419
32.0	121.344	134.978	148.548	162.051	175.501	208.800	241.700	274.256
32.5	129.292	143.335	157.311	171.213	185.064	219.340	253.199	286.700
33.0	137.820	152.276	166.662	180.966	195.222	230.487	265.318	299.779
33.5	146.959	161.831	176.631	191.340	206.004	242.271	278.086	313.523
34.0	156.740	172.031	187.247	202.366	217.441	254.720	291.533	327.961
34.5	167.196	182.908	198.543	214.074	229.563	267.866	305.688	343.125
35.0	178.360	194.493	210.549	226.498	242.401	281.738	320.584	359.046
35.5	190.264	206.820	223.299	239.668	255.986	296.369	336.252	375.757
36.0	202.942	219.922	236.825	253.618	270.351	311.792	352.724	393.292
36.5	216.430	233.834	251.161	268.383	285.528	328.038	370.034	
37.0	230.763	248.589	266.340	283.996	301.552	345.141	388.216	
37.5	245.976	264.222	282.399	300.493	318.456	363.137		
38.0	262.105	280.771	299.371	317.909	336.274	382.059		
38.5	279.188	298.270	317.293	336.281	355.042	401.943		
39.0	297.261	316.756	336.201	355.645	374.795			
39.5	316.363	336.268	356.131	376.040	395.569			
40.0	336.532	356.841	377.121	397.503				
40.5	357.807	378.515	399.209					

Table III(e)

## TEMPERATURE IN DEGREES KELVIN

DENSITY	70	75	80	85	90	95	100
16.5	91.488	102.095	112.668	123.209	133.732	144.175	154.586
17.0	94.984	106.064	117.103	128.120	139.118	150.010	160.881
17.5	98.595	110.162	121.683	133.184	144.662	156.032	167.374
18.0	102.328	114.396	126.413	138.408	150.374	162.243	174.068
18.5	106.191	118.773	131.301	143.801	156.265	168.648	180.965
19.0	110.192	123.301	136.355	149.372	162.345	175.251	188.072
19.5	114.341	127.991	141.583	155.130	168.626	182.062	195.398
20.0	118.648	132.852	146.994	161.086	175.117	189.089	202.952
20.5	123.123	137.894	152.600	167.249	181.832	196.345	210.746
21.0	127.778	143.130	158.412	173.632	188.780	203.841	218.793
21.5	132.624	148.570	164.442	180.246	195.975	211.592	227.107
22.0	137.674	154.228	170.702	187.105	203.427	219.613	235.704
22.5	142.941	160.118	177.206	194.220	211.151	227.918	244.598
23.0	148.439	166.252	183.969	201.608	219.159	236.524	253.806
23.5	154.182	172.646	191.006	209.281	227.466	245.447	263.344
24.0	160.185	179.315	198.331	217.256	236.086	254.703	273.228
24.5	166.465	186.275	205.962	225.549	245.036	264.309	283.474
25.0	173.036	193.543	213.916	234.176	254.332	274.279	294.096
25.5	179.918	201.135	222.209	243.156	263.991	284.630	305.109
26.0	187.126	209.069	230.859	252.506	274.033	295.375	316.526
26.5	194.680	217.364	239.886	262.247	284.477	306.528	328.359
27.0	202.598	226.039	249.307	272.396	295.343	318.101	340.619
27.5	210.900	235.113	259.143	282.977	306.655	330.105	353.313
28.0	219.606	244.606	269.413	294.009	318.437	342.549	366.449
28.5	228.738	254.539	280.137	305.514	330.712	355.439	380.031
29.0	238.315	264.934	291.336	317.518	343.508	368.782	394.061
29.5	248.362	275.811	303.031	330.042	356.853	382.579	
30.0	258.900	287.193	315.242	343.111	370.776	396.832	
30.5	269.954	299.104	327.992	356.752	385.310		
31.0	281.546	311.566	341.301	370.991	400.488		
31.5	293.703	324.604	355.193	385.855			
32.0	306.448	338.241	369.688	401.371			
32.5	319.809	352.503	384.809				
33.0	333.813	367.416	400.579				
33.5	348.485	383.004					
34.0	363.856	399.295					

(c) Tables IV, V and VI, Saturated Liquid Densities.

Results for para hydrogen from 17 -32°K are obtained by an iterative computer program for extrapolating above isotherms to the known vapor pressures. Results then are represented by

$$(\sigma - \sigma_c) = \sum_{m=1}^4 A_m (T_c - T)^{m/3} \quad (4)$$

where subscript c refers to critical conditions. Coefficients of table IV give computed results of tables V and VI. Data in the upper portion of table V and in all of table VI are from NBS RP 1932, adjusted to the 1955 NBS temperature scale.

(d) Table VII, giving some characteristic properties, is an aid for computational work.

## 1.2 Thermal Conductivity of Fluids

William J. Hall and R. L. Powell

The reference platinum resistance thermometer was calibrated at a number of points by intercomparison with an NBS-calibrated standard thermometer in an electrically-heated copper block. Resistance differences were taken between the thermometer being calibrated and a standard for which a detailed calibration table was available. A fifth order polynomial was fitted to the differences by a mean-least-squares approximation using a method of orthogonal functions. The interpolated differences were then combined with the standard table to obtain a detailed calibration table of the thermometer being calibrated. Since the hot and cold plate thermometers had already been compared to the reference thermometer under pressures up to 10 atm., the absolute calibrations of all three thermometers have now been established.

Table IV

Coefficients for Density-Temperature Relation  
at Saturation, Equation (4)

	<u>Para</u>	<u>Normal</u>
$A1 \cdot 10^3 =$	+ 6.267 5345	+ 6.646 6259
$A2 \cdot 10^3 =$	+ 1.497 3511	+ 1.026 5796
$A3 \cdot 10^3 =$	- 0.183 0690 3	+ 0.085 4904 60
$A4 \cdot 10^3 =$	- 0.020 6931 81	- 0.067 0342 34

Table V. Densities of Saturated Liquid Para Hydrogen

T, °K	V. P., atm.	$\sigma$ , exp.	$\sigma$ , calc.	$100 \frac{\Delta\sigma}{\sigma}$
13.803		.0382029	.0381998	.01
13.990		.0381286	.0381232	.01
14.000	.0778		.0381191	
14.990		.0377102	.0377030	.02
15.000	.1327		.0376987	
15.990		.0372633	.0372631	.00
16.000	.2130		.0372586	
16.991		.0367931	.0368012	-.02
17.992		.0362989	.0363159	-.05
18.993		.0357845	.0358047	-.06
19.995		.0352509	.0352642	-.04
17.000	.3250	.0368288	.0367970	.09
18.000	.4758	.0362733	.0363119	-.11
19.000	.6726	.0357945	.0358010	-.02
20.000	.9228	.0352753	.0352615	.04
20.268	1.0000		.0351115	
21.000	1.2341	.0346992	.0346898	.03
22.000	1.6142	.0341069	.0340821	.07
23.000	2.0714	.0334638	.0334330	.09
24.000	2.6135	.0327268	.0327363	-.03
25.000	3.2487	.0319869	.0319835	.01
26.000	3.9854	.0311607	.0311635	-.01
27.000	4.8315	.0302443	.0302610	-.06
28.000	5.7950	.0292449	.0292534	-.03
29.000	6.8890	.0281325	.0281060	.09
30.000	8.1210	.0267471	.0267588	-.04
31.000	9.5000	.0250788	.0250921	-.05
32.000	11.0470	.0227923	.0227821	.04
32.400			.0214352	
32.700			.0199780	
32.900			.0182832	
32.984	12.7700		.0152672	

Table VI Densities of Saturated Liquid Normal Hydrogen

<u>T, °K</u>	<u><math>\sigma</math>, exp.</u>	<u><math>\sigma</math>, calc.</u>	<u><math>\frac{\Delta\sigma}{100\sigma}</math></u>
13.947	.0383024	.0383038	-.00
13.990	.0382860	.0382861	-.00
14.000		.0382819	
14.990	.0378680	.0378653	.01
15.000		.0378609	
15.990	.0374230	.0374236	-.00
16.000		.0374190	
16.990	.0369530	.0369594	-.02
17.000		.0369546	
17.990	.0364610	.0364707	-.03
18.000		.0364656	
18.990	.0359500	.0359552	-.01
19.000		.0359498	
19.990	.0354200	.0354101	.03
20.000		.0354045	
20.380	.0352100	.0351889	.06
21.000		.0348263	
21.990	.0342340	.0342178	.05
22.000		.0342114	
23.000		.0335549	
23.990	.0328390	.0328580	-.06
24.000		.0328506	
25.000		.0320908	
25.990	.0312540	.0312737	-.06
26.000		.0312650	
27.000		.0303590	
27.990	.0293600	.0293629	-.01
28.000		.0293522	
29.000		.0282131	
29.990	.0269260	.0269034	.08
30.000		.0268889	
31.000		.0252776	
31.990	.0231420	.0231499	-.03
32.000		.0231238	
33.000		.0190252	
33.180		.0149365	



**Table VII. Some Characteristic Properties and Fixed Points**

		H. W. Woolley et al, 1948	H. J. Hoge et al, 1951	Ohio State Univ.	This Report	Penn. State Univ., 1954	NBS 1955
<b>HYDROGEN</b>							
<u>Triple Points</u>							
T, °K	p	13.81 <sub>3</sub>	13.81 <sub>3</sub>	13.84 <sub>5</sub>			13.80 <sub>3</sub>
	n	13.95 <sub>7</sub>					13.94 <sub>7</sub>
P, atm.	p	.0694 <sub>7</sub>	.0695	.0694 <sub>2</sub>			
	n	.0710 <sub>5</sub>		.0707 <sub>9</sub>			
v, cm <sup>3</sup>	p	26.176			26.178		
	n	26.108			26.107		
<u>Boiling Points</u>							
T, °K	p	20.27 <sub>3</sub>	20.27 <sub>8</sub>	20.26 <sub>1</sub>			20.26 <sub>8</sub>
	n	20.39 <sub>0</sub>		20.38 <sub>2</sub>		20.365 ±.005	20.380
v, cm <sup>3</sup>	p				28.481		
	n	28.401			28.418		
<u>Critical Points</u>							
T, °K	p		32.9 <sub>94</sub>				32.98 <sub>4</sub>
	n	33.19		33.24 <sub>4</sub>			33.18
P, atm.	p		12.7 <sub>70</sub>				
	n	12.98		12.797			
v, cm <sup>3</sup>	p		65.5				
	n	66.95					
<b>OXYGEN B.P.,</b>							
	°K.	90.190	90.190			90.154	90.180

Failure in another laboratory at CEL of a pressure gage containing hydrogen at a small fraction of full scale pressure has caused some concern. The Bourdon tube was steel and evidently had suffered hydrogen embrittlement. While hydrogen embrittlement in wet atmospheres or due to cathodic charging has been widely studied, embrittlement due to pure high-pressure hydrogen at room temperature is a rather rare occurrence, and little published information is available on it. The precision gages on this apparatus were furnished with steel Bourdon tubes, although the supplier realized that hydrogen service was involved. As a safety measure we have obtained replacement gages with beryllium-copper Bourdon tubes.

Since the last report the main efforts have been directed towards a detailed experimental testing of the apparatus with nitrogen as the standard conductivity fluid. This testing is required in order to check the internal consistency of the apparatus and to compare the measured values for the conductivity of fluid nitrogen with the results of earlier research reports from other laboratories. As a result of the test program, several systematic errors ranging from 0.1 to 1% have been discovered. Minor modifications were incorporated into the existing apparatus to remedy the observed faults. The internal inconsistencies are now less than 1%. However, disagreement with earlier published work was not substantially changed by any of the apparatus modifications. Therefore serious doubt is developing about the accuracy of published results based on either relative measurement methods or even absolute methods using a more complicated heat flow geometry. Auxiliary equipment has been added to the apparatus to improve efficiency, increase accuracy, and especially to reduce the time required for steady state conditions.

The tests for internal consistency are carried out with a wide range of measurement parameters: with the test fluid in either the gaseous or liquid phase, at low and medium pressures (1-50 atm. ), at various temperatures (70-120°K), at equilibrium (no thermal gradients), with various steady-state thermal gradients across the test space (0.5 to 2 deg K), and with spacers of various thicknesses (0.5 to 2mm) separating the hot plate from the cold plate. A change in the spacers requires a shut-down of the equipment, a dis-assembly of the apparatus, the actual change of spacers, and then re-assembly. Therefore all of the other consistency tests are carried out for a given spacing, then the spacers are changed and the tests repeated. At present the tests are nearly completed for the smallest spacer (0.5mm).

The first few runs showed one systematic error of about 1%; i. e. , the apparent conductivity was a function of the thermal gradient. The fault was traced to readings from no gradient runs that are required for zero corrections to the regular gradient runs. The basic source of the error appeared to be the second auxiliary guard within the pressure cell. It was changed from being in thermal contact with the outer shell to thermal contact with the first guard. At the same time, several of the thermometer leads were changed to give greater thermal tempering just before the thermometer capsules. The apparent dependence on thermal gradient was greatly reduced by the modifications. Since then no systematic errors greater than 0.1% have been observed: the total error seems to be caused primarily by uncertainties in potentiometer readings and slight thermal drifts during the actual runs.

The consistency check runs for the gas phase (e. g. 1 atm at 126°K, 0.5mm spacer, and with 0, 3/4, 1, and 2 deg K gradients)

show reasonable agreement with earlier work: our results are intermediate between the others; higher than Ziebland's, lower than Uhler's. The disagreement with either author is about 1-3%. The comparisons for the liquid phase are quite different however. We are about 12% higher than Ziebland, 5% higher than Powers, et al. At present this disagreement does not appear to be resolvable. None of the alterations of the apparatus have changed the results on liquids substantially. The errors in previous work are most likely caused by either uncertainties in the corrections for convection or by a thin film of gas on the hot plate or wire.

The main change in the apparatus was the one mentioned above, the revision of the auxiliary guard. However, the approach to steady state has been made easier by the installation of a manostat above the liquid refrigerant. The system temperature was previously sensitive to the barometric pressure above the refrigerant. The drifting was particularly bad whenever the barometric pressure changed rapidly. The change in temperature of the basic unit caused by the drift of the refrigerant temperature has now been greatly reduced and is no longer a problem.

## 2. Cryogenic Instrumentation

J. Macinko, P. Smelser, C. E. Miller, R. C. Muhlenhaupt and R. B. Jacobs

During the past reporting period the pressure transducer calibration unit was put into operation and a program of calibration of commercial instruments was initiated. Fabrication of the first temperature transducer test unit was completed and the second unit started. The first draft of the instrumentation survey was completed and copies are enclosed.

### Pressure Transducers

Modifications of the calibration unit and control equipment were made during this period. The major change was in the temperature measurement of transducers under test. The original design called for a small vapor-pressure thermometer. Due to a number of inconveniences related to preparing the bulb for different cryogenic fluids and problems in recording output, platinum resistance thermometers have been substituted for vapor bulbs. The pressurizing line was redesigned to provide greater control of the gas input and a more stable signal to the precision pressure gages.

Transducers are now being evaluated from 20°K to 300°K. The test data are being compiled to be issued in the form of graphs and charts as a supplement to the instrumentation survey.

### Temperature Sensor Test Unit

The assembly of the first test apparatus (Figure 1, Second Quarterly Report) to determine temperature response characteristics has been completed and preliminary pumpdown and cold shock tests are being run. Minor structural changes have been made to facilitate machining and assembly but the overall design is still as described in the above reference. Associated instrumentation for this test apparatus has been fabricated and the entire unit is now mounted at the test site.

Design studies are continuing on the adiabatic compression apparatus which is to provide a step-function temperature change to sensors submerged in liquid. Although the principle is quite simple, numerous problems have arisen in each scheme devised to carry out the compression process. Due to the combination of high pressure (3000 psi) and low temperature requirements, a satisfactory valve or other type of release mechanism was a problem which delayed the

final design. The necessity of retaining a small expansion ratio presents problems in fill and vent line design, instrument lines, and sensor installation and removal. However, even with these problems the system using gas pressurization of a liquid volume appears to be more feasible than a piston system. Since a mechanically driven piston would have a fixed displacement, elimination or control of the gas in the liquid volume undergoing compression is necessary in order to control the compression ratio. The use of a pneumatic system not only eliminates the need for this control but eliminates the need for a piston as well. Fabrication of the high pressure vessels has started and the detailed design of the final apparatus will be completed as soon as the high pressure units prove to be functional.

#### Liquid Level

A program to evaluate various commercial point sensors and continuous reading liquid level gages has been initiated. The first stage of the program has been concerned with investigating ideas for a primary standard. Figure 1 is one such idea currently under evaluation. A model similar to the one shown has been built.

Gas at extremely low positive pressure ( $0.02-0.05'' \text{H}_2\text{O}$ ) flows through the probe and is discharged at the lower end. The probe is mounted on an adjustable platform which provides a means of aligning the probe perpendicular to the liquid surface. A vernier arrangement is used to raise and lower the probe. The movement is followed by a dial indicator which will measure displacements to the nearest  $1/1000''$ . When the probe end comes in contact with the liquid surface erratic fluctuations of pressure occur and are observed on a sensitive pressure gage. Present indications are that liquid level measurements can be accurately made within  $0.01''$  for quiescent

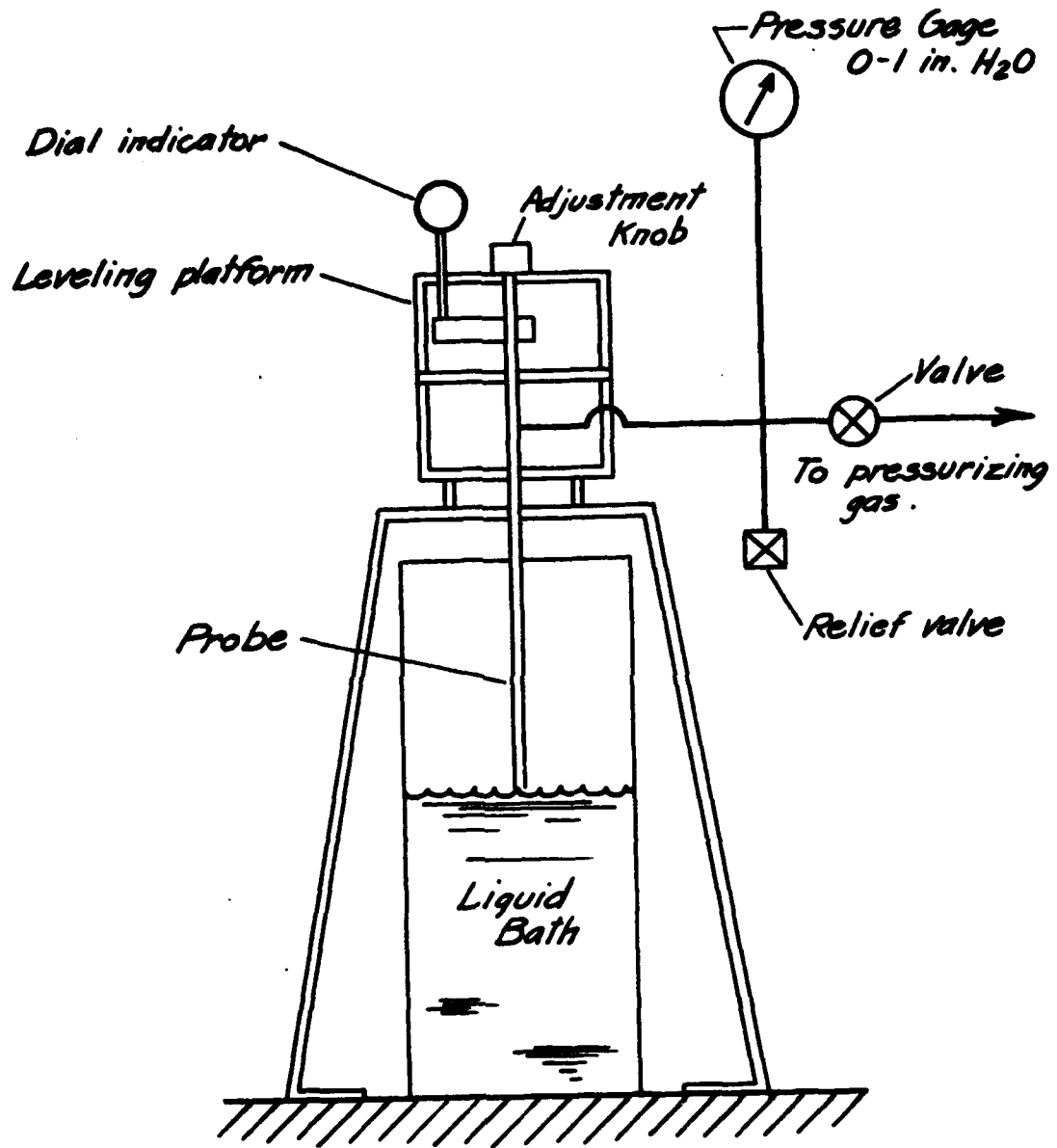


Fig. 1 Liquid level gage.

cryogenic fluids. The accuracy of measurements in violently boiling fluids has not been determined.

#### Forced Vibration Densitometer Studies

A preliminary evaluation of the densitometer has been completed. Figure 2 is a photograph of the experimental model and associated control and recording equipment. A more detailed view of the instrument is presented in Figure 3. The existing model was designed primarily to evaluate the principle of operation. Because of the difficulties in fabricating a model suitable for cryogenic applications, tests have been conducted exclusively on ambient temperature fluids. Furthermore, due to an unforeseen pressure sensitivity characteristic of the bellows (i. e. , the change in apparent stiffness resulting from a change in internal pressure) it was necessary to restrict tests to no-flow conditions. To maintain a relatively constant pressure in the flow passage two reservoirs, located on each side of the densitometer, were used. Fluids were selected which had specific gravities ranging from 0.72 to 1.35. During these tests the forcing frequency was held at 12 cps. Results from a series of calibration tests showed the model capable of determining densities to within  $\pm 1\%$ . Figure 4 shows a typical calibration curve.

One additional test was conducted to provide information on the instrument's ability to measure bulk densities of two phase fluids. The test consisted of draining known quantities of water from the vibrated passage and recording the densitometer output. Figure 5 presents the results of that test. Although a more thorough study of two phase measurements is needed, the results obtained are encouraging.





Figure 2

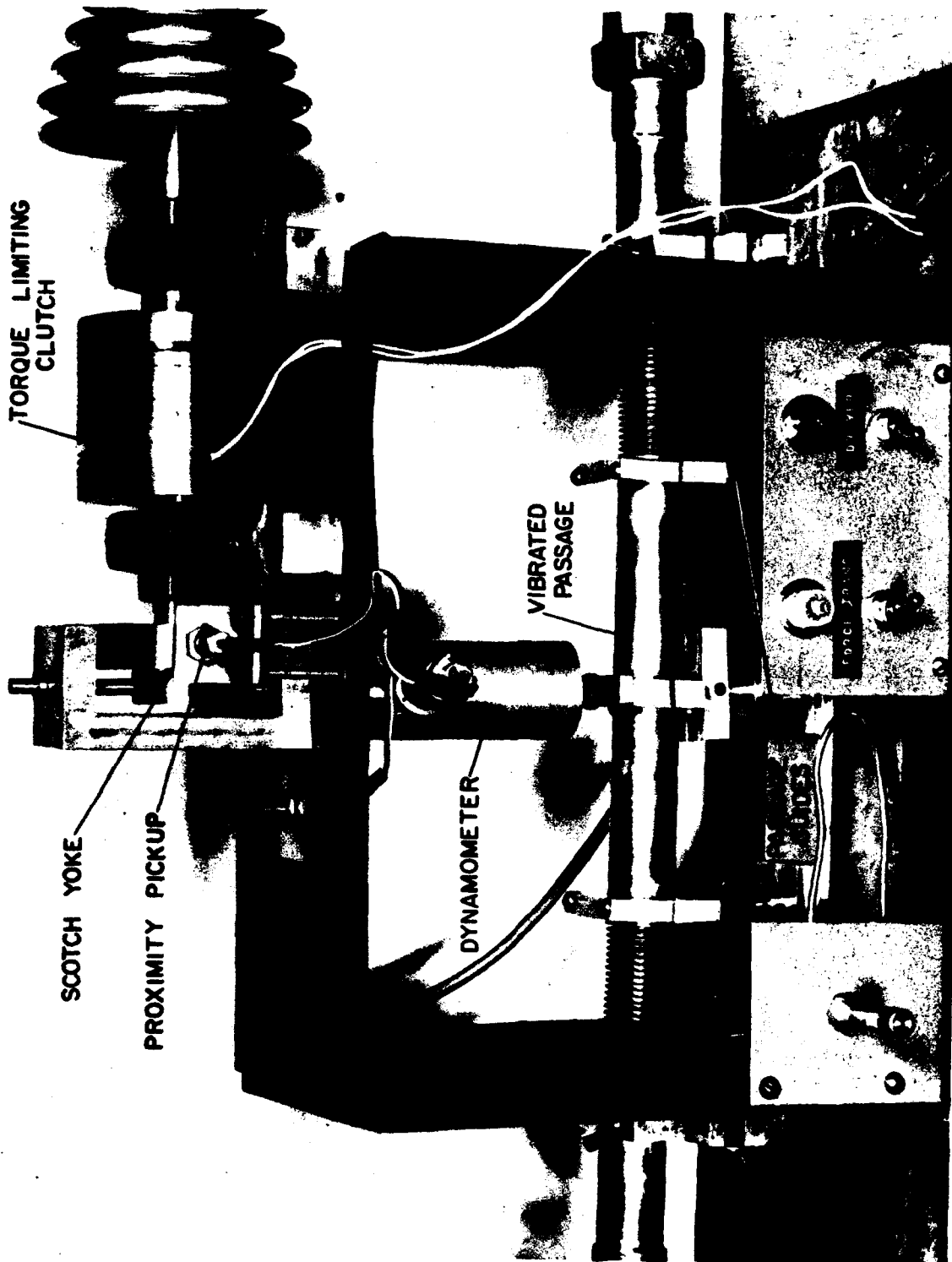


Figure 3

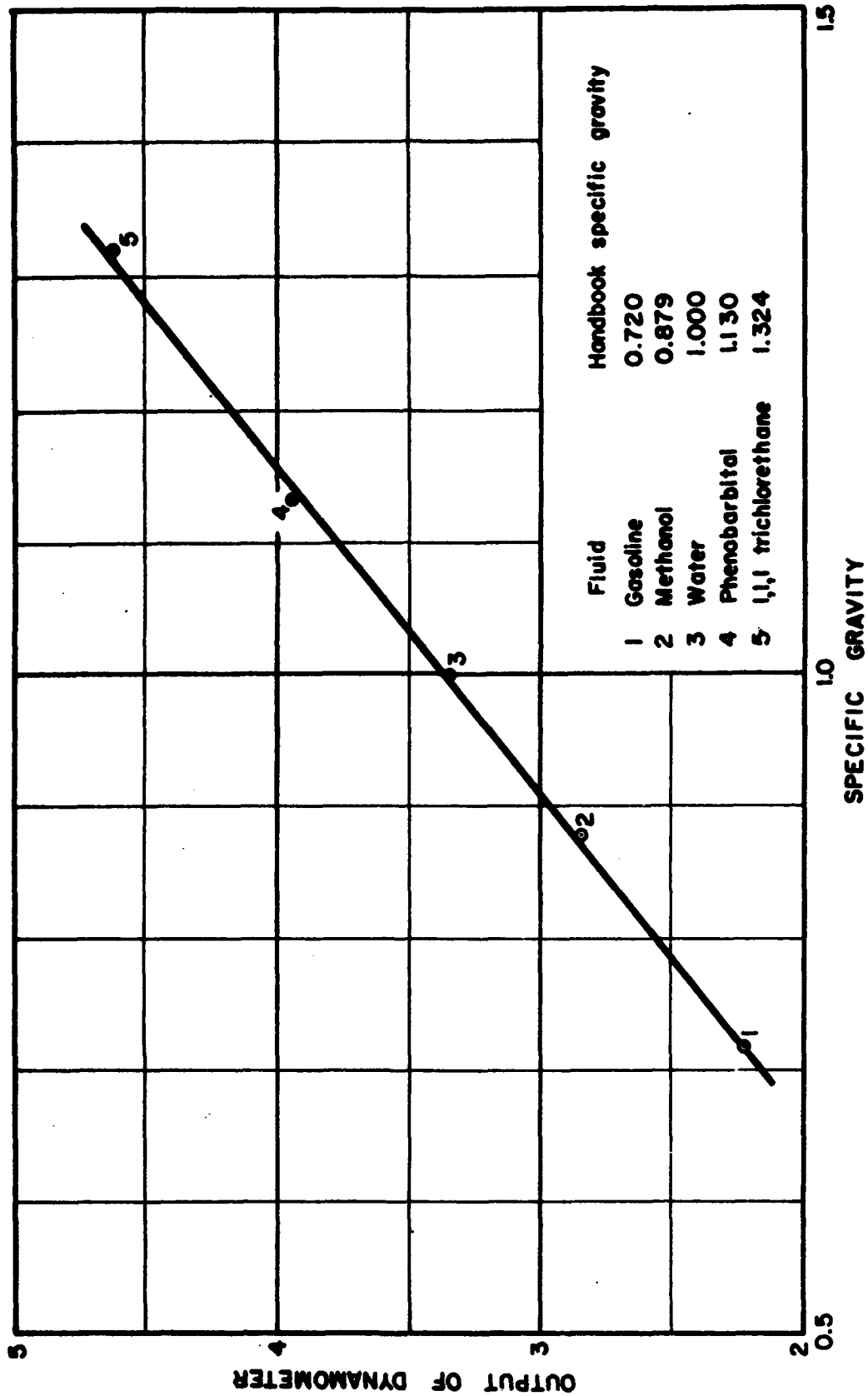


Figure 4 · DENSITOMETER CALIBRATION CURVE

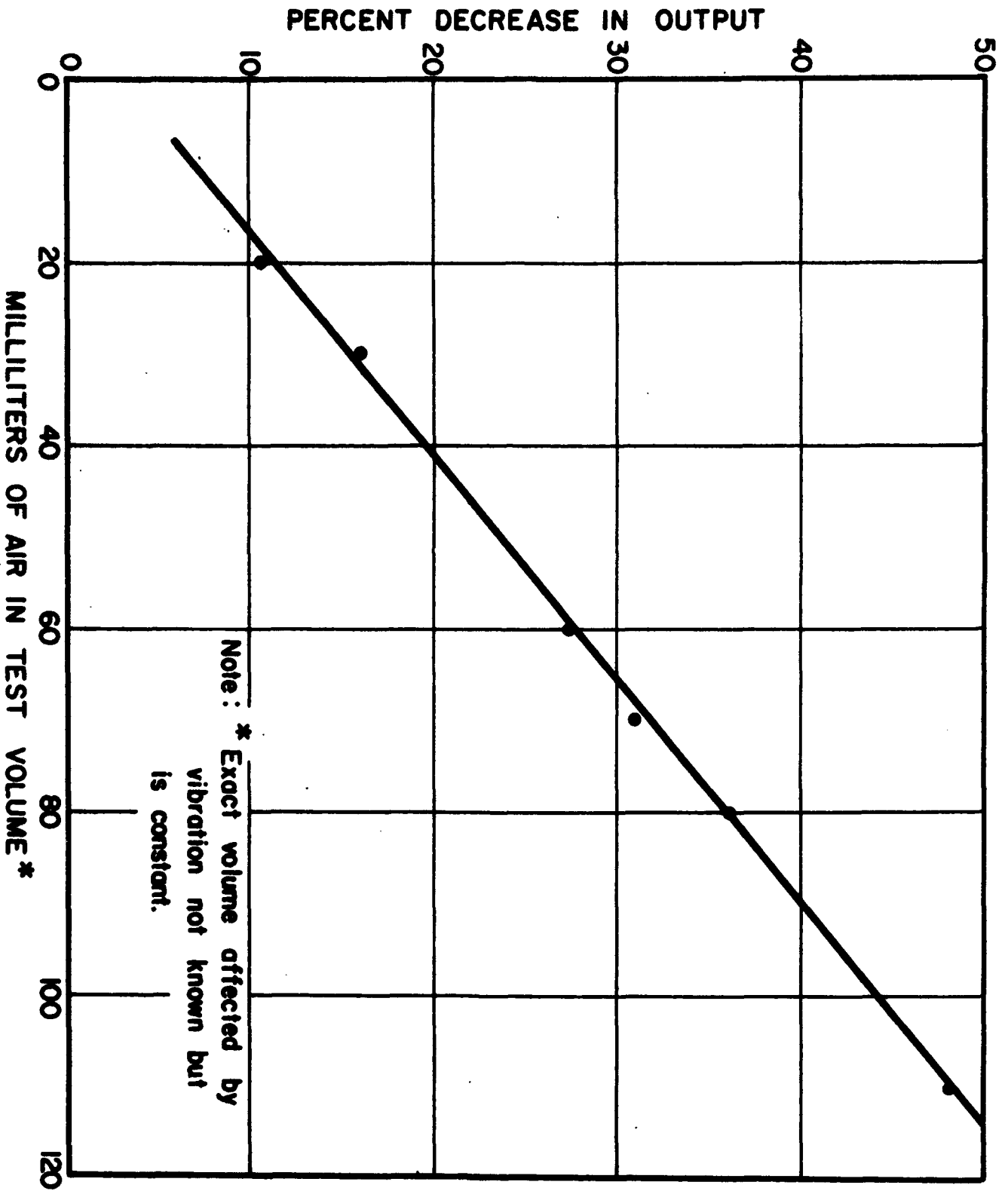


Figure 5 · BULK DENSITY FOR WATER AND AIR MIXTURE

A second model, suitable for low temperature applications, has been designed and is presently under construction. A cutaway view of this model is presented in Figure 6. The pressure sensitivity of the bellows is compensated for by external pressurization. A pressurized housing encloses both the couplings and the force gage. Pressurization of the housing can be accomplished by two independent methods, depending on the thermodynamic state of the cryogenic fluid being measured. When the fluid is in a saturated state, a small line joining the flow passage to the housing is opened, allowing vapor formed in the line to discharge into the housing. Vapor will continue to fill the housing until pressure equilibrium is achieved. For the case involving subcooled liquids, pressurization is accomplished by using a separate gas supply. This, however, requires a pressure regulator to null the pressure differential across the bellows. Numerous refinements and modifications have been incorporated into the design of this model. Special considerations have been given to the vibration exciter and vacuum insulation system. In addition, the densitometer's readout system is to be improved over the former system.

Plans for the future call for evaluating the densitometer at cryogenic temperatures under both single and two phase flow.

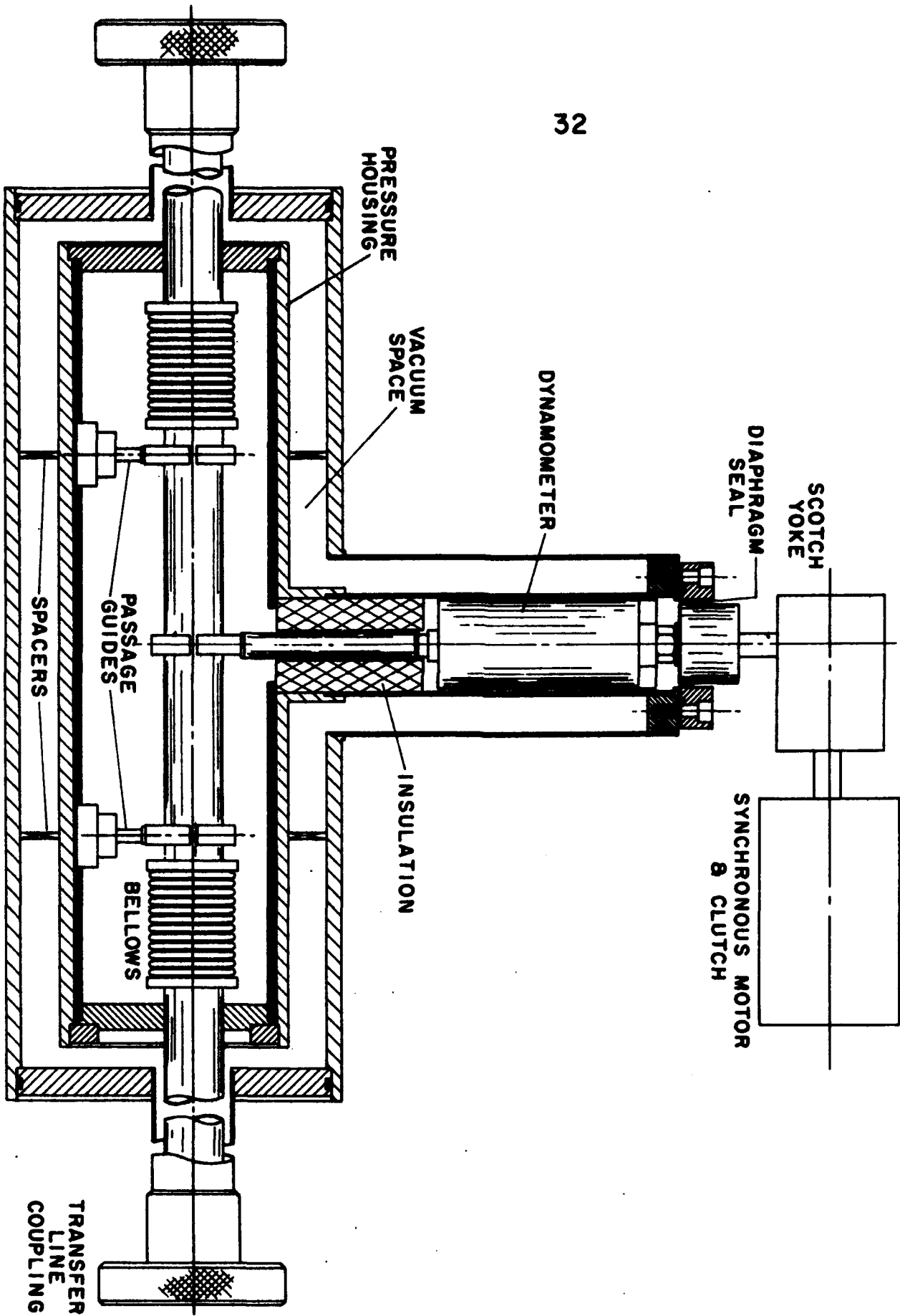


Figure 6. Densitometer

### 3. Cryogenic Design Principles and Materials Utilization

D. B. Chelton, L. E. Scott, J. A. Brennan and B. W. Birmingham

General assistance on Project Centaur has continued during the reporting period. Although, primary assistance efforts were with General Dynamics-Astronautics (GDA), several contacts were made with co-contractors and sub-contractors associated with the program.

Emphasis on the GDA portion of the program has been focused on the development of cryogenic seals for the liquid hydrogen fuel line flange application, on the cryogenic testing of ball bearings operating in hydrogen gas for the zero-gravity centrifugal vent device and on insulation problems associated with the flight vehicle and the propulsion test vehicle located at Sycamore Test Facility.

A detailed discussion of some of the technical problems that have been considered is given below. In addition, assistance was given to GDA personnel on other matters of cryogenic importance such as properties of materials, instrumentation, liquid oxygen density, gas detection, and thermometry.

A total of three visits were made to GDA by NBS personnel and several trips by GDA personnel were made to NBS. Also, a number of telephone conversations were made to assist GDA personnel. In addition to these direct contacts, visits were made to NASA-MSFC, Pratt and Whitney Aircraft, AMR, Rocketdyne, and Douglas Aircraft Company. Assistance to these organizations was in the nature of consultation on general cryogenic problems.

To date, very little assistance has been given to the Rover program. Although several contacts have been made with LASL personnel, a course of action has not yet been formulated. It is

hoped that contributions can be made in this area during the next reporting period.

### Narmco Mechanical Properties Testing

General assistance has been given to Narmco Industries, Inc., San Diego, California on a materials testing program they are undertaking for NASA. The test program is devoted to the mechanical properties of non-metallic materials and adhesives. Temperatures down to that of liquid hydrogen will be included.

At the present time, the test facility is under design by Stearns-Roger Manufacturing Company, Denver, Colorado. Construction of the facility is also planned by that company. In this phase, our assistance has been to review the design from a cryogenic and safety standpoint. Several meetings have been held with the organizations involved. The basic NBS-CEL designs for test cryostats will be incorporated in the facility whenever applicable.

A Narmco representative has been present for a period of one week to witness mechanical properties tests at our laboratory. This was a brief effort at operator orientation. Tentative arrangements have been made for longer orientation periods.

#### 3.1 Propellant Tank Insulation

The spray foam insulation applied to the Propellant Test Vehicle (PTV) at the Sycamore site developed typical failures on the first cooldown. The installation and initial failures occurred during the previous reporting period. These failures continued to propagate during tests conducted during the present period.

The large radial and longitudinal cracks induced by thermal stresses have been continually patched with a flexible foam and covered with tape to provide a vapor barrier. In addition, since it



appeared to be separating from the tank surface, metal bands were used to secure the insulation.

A solution to the immediate and long range problem has not been readily apparent. Several recommendations for a development program have however, been made to GDA personnel. Considerable emphasis on insulation development, not necessarily limited to foams, has been encouraged. A meeting of several interested groups was held in an effort to arrive at an interim solution. From the meeting, three possible procedures were suggested. These are as follows:

(1) Cover the existing insulation with Mylar or a wet lay up of fiberglass and use a helium purge. This is an interim solution so the tank can be used for the current tests and will not interfere with the schedule. It is hoped that the outer layer can be made sufficiently tight to inhibit the formation of liquid air and reduce the safety hazard.

(2) V. Gray, NASA, suggested placing Styrofoam blocks (unbonded) around the container and sealing it with a wet lay up of fiberglass. The entire space is then purged with helium. R. H. Kropschot (NBS) suggested using foam glass because of its closed cell nature and, in addition, it is inert.

(3) Linde suggested the use of fiberglass covered with Mylar and evacuated. They expected a conductivity of approximately  $50 \frac{\mu \text{ watts}}{\text{cm}^2 \text{ } ^\circ \text{K}}$ . They have insulated small smooth containers with this technique with some success. It was questioned by the group whether this technique could be applied to the cruiser tank because of the large number of pipes and wires coming out through the Mylar seal.

Recommendations (2) and (3) are possible insulation schemes to be placed on the existing tank when it is removed from the Sycamore

site and taken to Edwards 1 - 1 site. If another tank of this type was to be built, serious consideration would be given to high vacuum or vacuum powder insulation.

Suggestion 1 was adopted by GDA for continuation of the tests at Sycamore. The plastic enclosure with a helium purge was reasonably effective. The PTV has now been moved for the installation at the Edwards Test Site. Modifications have been made according to suggestion 2. A high vacuum insulated container is presently being considered for additional PTV at Edwards.

A small experimental program is being conducted at CEL in an effort to evaluate a few relatively new epoxy and polyurethane foams. Several selected foams have been sprayed on flat "pans" for preliminary evaluation. To date, none have been completely successful - cracks usually appearing upon repeated thermal cycling to liquid nitrogen temperature. The most promising sample has been one in which a fiberglass - epoxy layer was applied to the metal surface prior to spraying with epoxy foam. If successful materials or combinations of materials can be discovered they will be applied to several available 30 liter vessels.

### 3.2 Flange Seals for Centaur Cryogenic Ducting

The Centaur vehicle uses several flanged joints in the cryogenic fuel ducting. These joints have been a source of intermittent leakage problems resulting from gasket failure. NBS-CEL has been active in the development of elastomers and Mylar plastic as cryogenic sealing materials and has suggested elastomer O-rings as a possible superior gasket for this application. A test program, using Centaur hardware was initiated by both NBS and GDA. This report concludes the NBS portion of this program.

### 3.2.1 Previous Experiments

Previous experiments<sup>[1]</sup> have shown that an absolute low temperature static seal can be made using elastomers and Mylar film in several special assemblies. Mylar<sup>[R]</sup> film was used in gasket form and was compressed against a flat surface with a compression ring of small diameter machined on the opposite plate. The elastomer O-rings were sealed in a tongue and groove geometry between rigid plates. Compounds (of elastomers) found acceptable were those utilizing the basic polymers of Viton-A, natural rubber, nitrile rubber, and neoprene.

O-rings of these compounds made excellent seals when compressed 80% or more, without lubrication, in a groove designed for a slight (min. of 5%) volume compression at the required squeeze. All seals leaked less than  $3 \times 10^{-4}$  standard cc of helium per hour, at all pressures from atmospheric to 1000 psig and at all temperatures from ambient to 20°K. In addition, some seals were subjected to vibration tests and retested with identical results. The seals described above require up to 40 ft. lbs. on each of six 3/8 inch 24 N. F. thread bolts for a 1/8" x 1" O-ring. Loading is reduced with the use of the Mylar gasket seal which requires a minimum 70% thickness compression (from 10 to 3 mils). The high stress concentration of the compression ring required a loading of only 5 ft. lbs. on each of six 1/4 inch 28 N. F. thread bolts for a 1" diameter gasket.

[1] Paper D-6, 1960 Cryogenic Engineering Conference, by Weitzel, et al.

[R] Du Pont Registered trademark

The elastomer O-ring concept has been explored in two additional ways. These are the use of O-rings of small cross-section in the tongue and groove flange design and the use of unconfined O-rings between flat plates. Viton A<sup>[R]</sup> O-rings (only material tested) of 1/16 inch cross-section diameter have made satisfactory seals in a tongue and groove flange design and required about half as much bolt loading as 1/8 inch O-rings of the same material. Hypalon, neoprene, and Viton-A have made excellent low temperature seals when squeezed to 20% of their original thickness between two flat flanges. Both 1/8 inch and 1/16 inch unconfined O-rings have been successful with approximately half the loading required for a confined O-ring of the same cross-section.

### 3.2.2 Experimental Details

A detailed study of the possibilities of flat flange seals was undertaken in connection with requirements of the Centaur missile program. A number of flanges supplied or specified by General Dynamics Astronautics were studied, using the method shown schematically in Figure 7. The flange and seal assembly was surrounded by a vacuum jacket which was maintained by a roughing pump and a helium leak detector. A stainless steel tube which passed from the seal assembly through the top plate of the vacuum can could be used to introduce either helium pressure or refrigerant (liquid N<sub>2</sub> or H<sub>2</sub>) into the seal assembly. Thus the seal could be tested at any desired pressure, either before, after, or during cooldown, and any helium escaping into the vacuum space would be immediately observed by the leak detector.

Leak rates greater than 0.5 cc per hour exceeded the capacity of the leak detector. These were evaluated by removing the

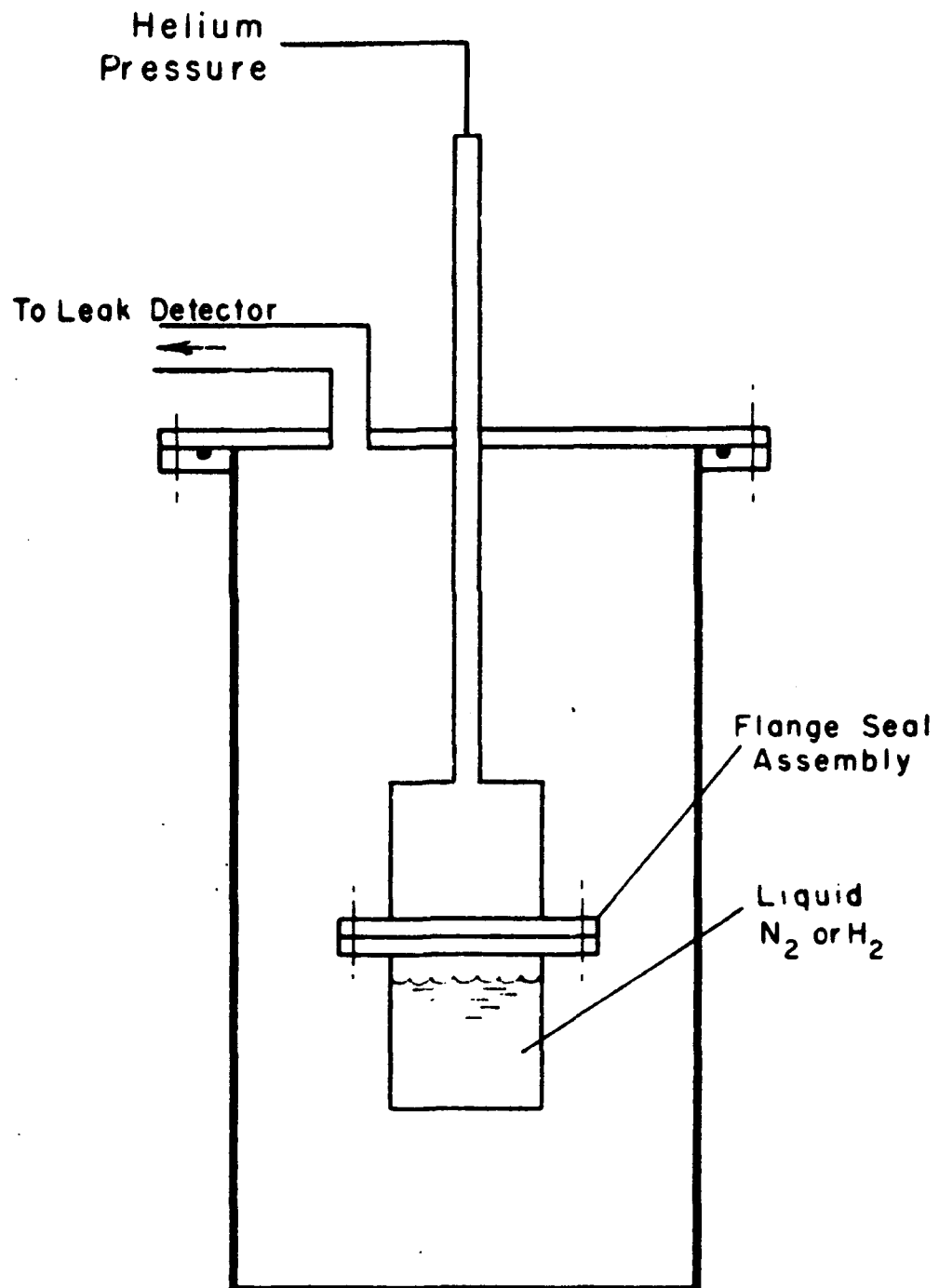


FIGURE 7

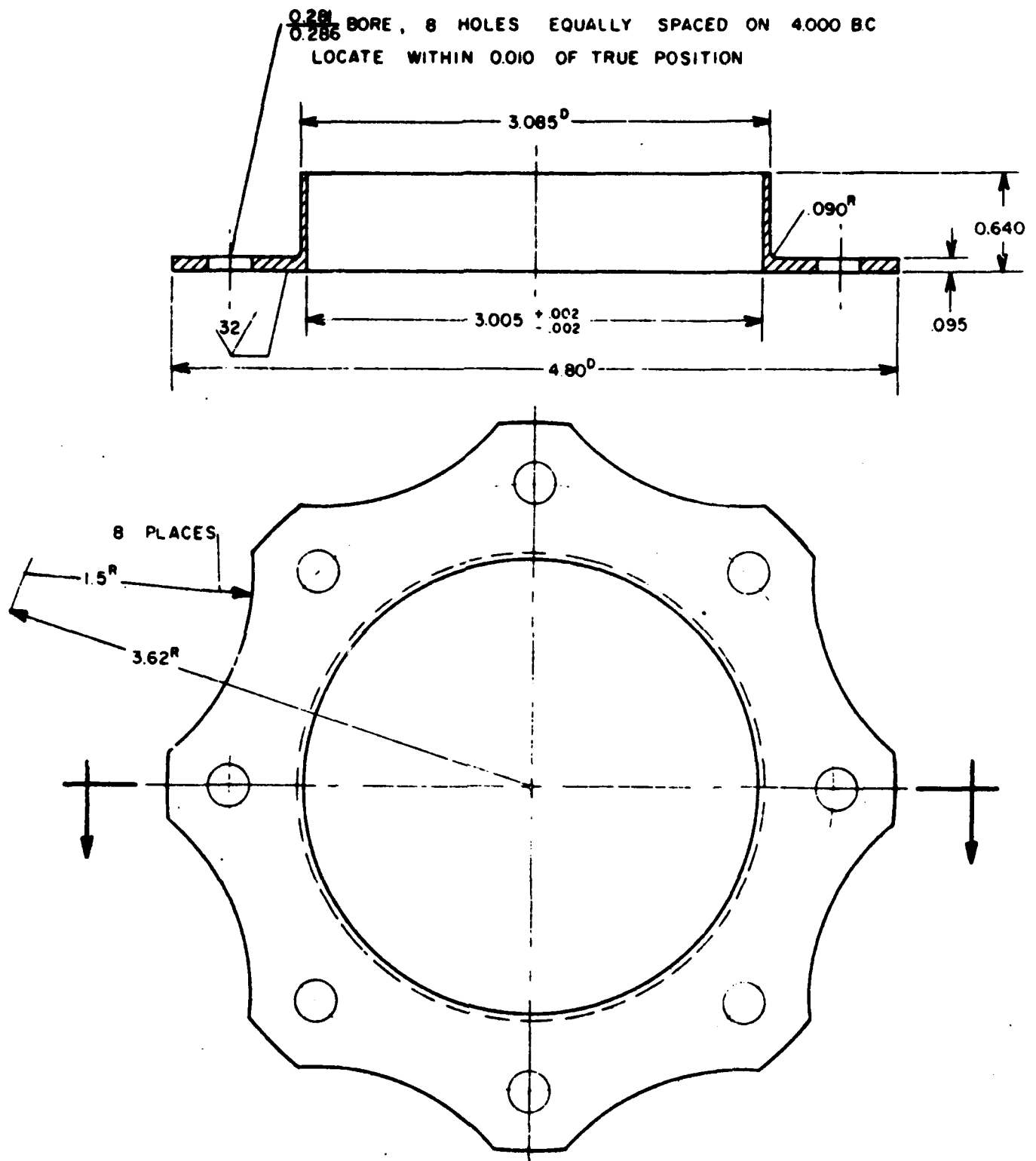
Schematic of Seal Test Fixture

vacuum jacket and submerging the flange assembly in water or liquid nitrogen. The inside of the assembly was then pressurized with helium gas, sealed off, and the leak rate determined by observing the rate of pressure decay.

Test procedure consisted of cleaning the flange faces with trichloroethylene and installing the O-ring as received from the manufacturer. Compression was by stepwise cross-tightening of the flange bolts with the aid of a calibrated torque wrench. The threads of the bolts were kept lubricated with a molybdenum disulfide aerosol dispersion. The assembly was then placed in the vacuum can and the latter evacuated. The seal was checked at room temperature by purging out the air and then pressurizing with helium gas. Success of the seal was determined by a zero reading on the most sensitive scale of the leak detector, which meant a leak of less than  $3 \times 10^{-4}$  standard cc of helium per hour. Higher leak rates were determined by reading the leak detector scales or by pressure decay as mentioned above. Some of the seals were subjected to a series of 25 temperature cycles by removing the flange assembly from the vacuum can and alternately submerging it in liquid nitrogen and hot water. The assembly was then placed in the vacuum can and again tested for leaks. Rapid pressure cycling was accomplished by means of an off-on switch which operated a three-port solenoid valve.

### 3.2.3 Extreme Lightweight Flange

One application involving airborne hardware for the Centaur missile required that weight be kept to an absolute minimum. The flange proposed by Astronautics for this application is shown in Figure 8. The flange, which fits a 3" O.D. pipe, was made of type



Scale: Full

Note: Fractional tolerance  $\pm 1/64$ 

Material: 304 SS.

Decimal tolerance  $\pm .010$ , except  
where noted.**FIGURE 8****Extreme Lightweight Flange**

304 stainless steel with faces only .095" thick. There were eight holes for 1/4" bolts, equally spaced on a 4" bolt circle, and further reduction in weight was made by cutting out scallops of material between the bolt holes. The various methods which were tested for sealing pairs of these flanges are listed in Table 8. Three seal materials were used. These were Parco Hypalon<sup>[R]</sup> Compound 921, durometer 50, Parco Neoprenes Compound 307, durometer 50; and 308, durometer 30; and Mylar<sup>[R]</sup> film. Hypalon and Neoprene were used as O-rings in two thicknesses, .070" and .103". On some of the O-ring tests a short stainless steel sleeve was fitted snugly inside the flange to hold the O-ring in position and prevent inward extrusion. A radial cross section of a flange assembly using such a sleeve is shown in Figure 9. Mylar film was used by making "sandwich" of two flat Mylar gaskets separated by an O-ring shaped compression ring made of nichrome wire. The ends of the wire were tapered and silver soldered to make a smooth joint.

No really satisfactory method for sealing these extremely light-weight flanges was found. Some of the seals held at room temperature, and one Hypalon O-ring used with the sleeve was leak detector tight at 76°K. But even this successful seal was probably borderline since a second attempt of the same kind failed when cooled to 76°K.

The reason for failure of these seals was lack of sufficient stiffness in the flange faces. Attempts to overcome this weakness by various schemes, as noted under "comments" in Table 8 were not successful. It is felt that the flange could be designed for greater rigidity with little or no increase in weight. In the present design there is a minimum stiffness between bolts, which is precisely where



TABLE 8. Static Seal Tests Using Convair Astronetics  
Extreme Lightweight Flange

Test No.	Type of Seal	Size of Seal	Torque on Eight 1/4" N.F. Bolts	Comments and Results*
14.	Parco Hypalon <sup>R</sup> 921-50 O-ring	3.480" I.D. .070" W	125 in-lb	O.K. warm. Pressure cycled 10 times. Leak rate cold approximately .5 cc/hr
23.	ditto	3.239" I.D. .070" W	125 in-lb	Flanges backed up by carbon steel ring under bolts. Used O-ring retaining sleeve. O.K. warm, leaked cold.
25	ditto	3.489" I.D. .070" W	125 in-lb	Flanges backed up. No sleeve. O.K. warm and at 1 atm cold. Leaked at 50 psig.
27	ditto	3.489" I.D.	125 in-lb	Flanges backed up and .005" shims under backing between bolts. O.K. warm, leaked cold.
31	ditto	3.239" I.D. .070" W	125 in-lb	Flanges given reverse bend before bolting. O.K. warm, leaked while cooling.

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\* O.K. means leak rate less than  $3 \times 10^{-4}$  cc/hr of helium at 160 psig.

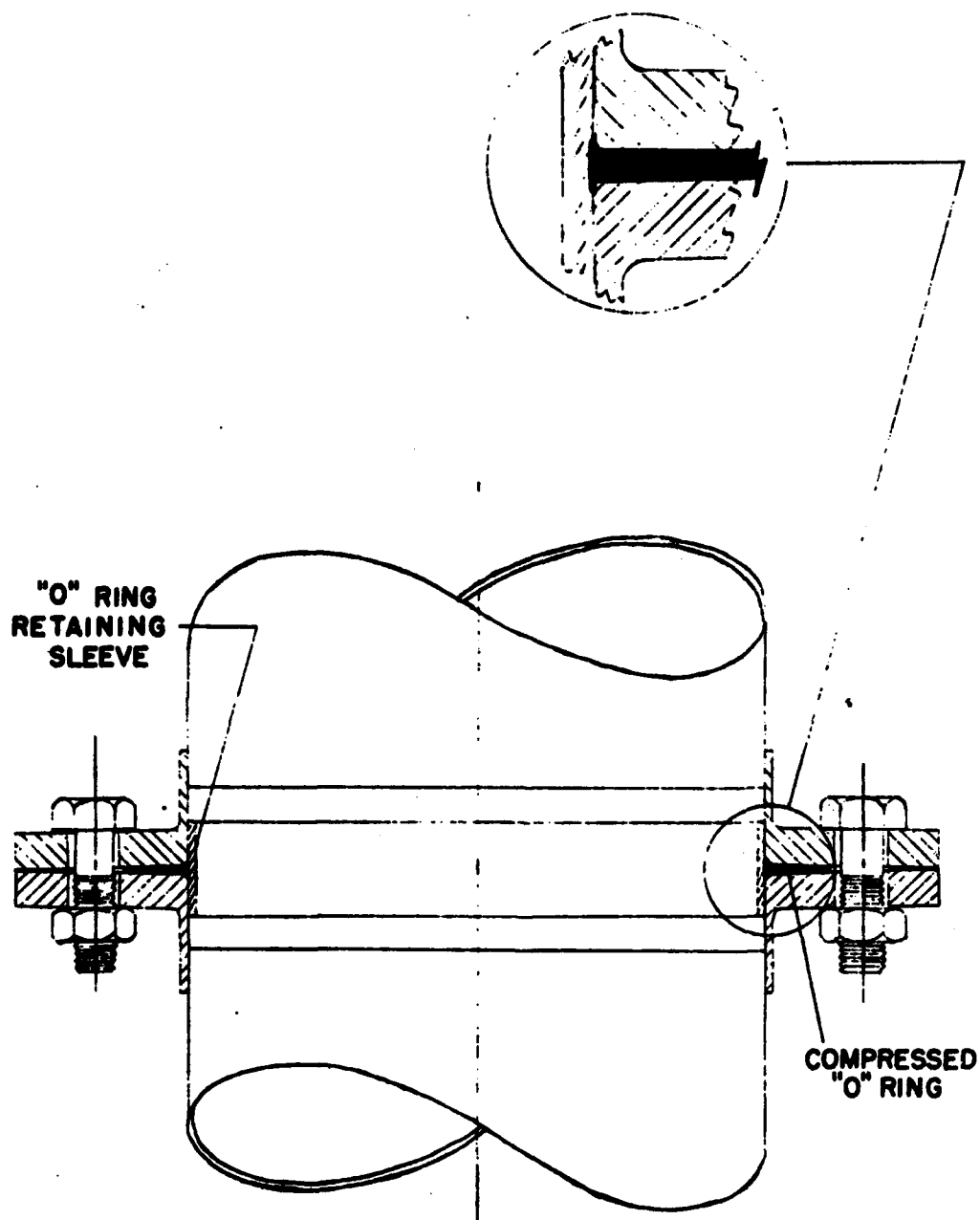
(Table 8. Continued on next page)

Test No.	Type of Seal	Size of Seal	Torque on Eight 1/4" N. F. Bolts	Comments and Results
43.	Parco Hypalon 921-50 O-ring	3. 239" I. D. .070" W	150 in-lb	One lightweight flange vs. heavy aluminum. Used sleeve but O-ring loose on sleeve. O.K. warm, leaked while cooling.
45.	ditto	2. 864" I. D. .070" W	ditto	One lightweight flange vs. heavy aluminum. O-ring stretched over sleeve. <u>O.K. warm and cold.</u>
47.	ditto	ditto	ditto	Repeat of 45. Leaked cold. Increased torque to 165 in-lb. Still leaked at 76°K.
16.	ditto	3. 237" I. D. .103" W	125 in-lb	O.K. warm, leaked while cooling. At 76°K leaked approx. 180 liters/hr. 44
24.	ditto	3. 237" I. D. .103" W	ditto	Same configuration as 23. O.K. warm, leaked while cooling.
26.	ditto	3. 489" I. D. .103" W	ditto	Same configuration as 25. O.K. warm, leaked while cooling.
15.	Parco Neoprene 308-30 O-ring	3. 237" I. D. .103" W	ditto	Soft - 30 durometer. Leaked warm.
28.	Parco Neoprene 307-50 O-ring	3. 489" I. D. .070" W	ditto	Same configuration as 25. Failed warm. Non-uniform compression.

(Table 8. Continued on next page)

<u>Test No.</u>	<u>Type of Seal</u>	<u>Size of Seal</u>	<u>Torque on Eight 1/4" N.F. Bolts</u>	<u>Comments and Results</u>
29.	Parco Neoprene 307-50 O-ring	3. 239" I.D. .070" W	125 in-lb	Same configuration as 27. Failed warm. Non-uniform compression.
30.	ditto	3. 239" I.D. .070"W	125 in-lb	Same configuration as 27, but used sleeve. O.K. warm, leaked while cooling.
13.	.0075" Mylar <sup>R</sup> .065" Nichrome wire	Wire 3 1/4" I.D.	80 in-lb	Braced with .040" wire ring outside bolts. Leaked warm - no compression between bolts.
17.	.0075" Mylar .040" Nichrome	Wire 3 1/4" I.D.	60 in-lb	Same configuration as 13. Again leaked warm.
21.	.012 Mylar .050 Nichrome	Wire 3 1/8" I.D.	60 in-lb	Uneven compression. Leaked warm.
20.	ditto	Wire 3 1/4" I.D.	60 in-lb	Somewhat better compression. Leaked in H <sub>2</sub> O at 10 Psig.
34.	.005" Mylar .040" Nichrome	Wire 3 1/4" I.D.	100-125 in-lb	Flanges backed up by carbon steel rings under bolts. Leak rate 0.2 cc/hr at 76°K, 50 Psig.

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**FIGURE 9**

**Typical Flange Assembly  
With "O" Ring Retaining Sleeve**

maximum stresses occur. A rib between the bolt holes would greatly improve the flange characteristics.

### 3.2.4 Successful Flat Flanges

Additional flanges specified for the Centaur program are shown in Figures 10 and 11. These flanges, for 2.5" and 3" O. D. pipelines respectively, were made of stainless steel with flange faces .200" thick. The smaller flange was drilled for ten 1/4" NF 18-8 stainless steel bolts on a 3.345" bolt circle, and the inside corner was given additional strength. The larger flange lacked this reinforced corner and used only 8 bolts (same bolts as the lightweight flange) on a 4" bolt circle.

Certain parts of the Centaur fuel handling equipment required a transition from stainless steel to aluminum. The flanges shown in Figures 10 and 11 were therefore duplicated from 6061, T6 aluminum, except that the flange faces were made .300" thick instead of .200". These were tested, both before and after anodizing to a black hardcoat finish, by mating with the corresponding stainless steel flanges. Except for the anodizing treatment, which was tested both polished and unpolished, all flange surfaces were used in the as-machined condition.

Table 9 gives the results of testing various combinations of these flanges with seals of Neoprene, Hypalon<sup>[R]</sup>, Viton<sup>[R]</sup>, Mylar<sup>[R]</sup>, and Teflon<sup>[R]</sup>.

Successful seals were obtained with all of the flange combinations. The Hypalon compound seemed to be somewhat more reliable than the Neoprene, and was tested more extensively. The Viton compound was successful in both of the tests for which it was used.

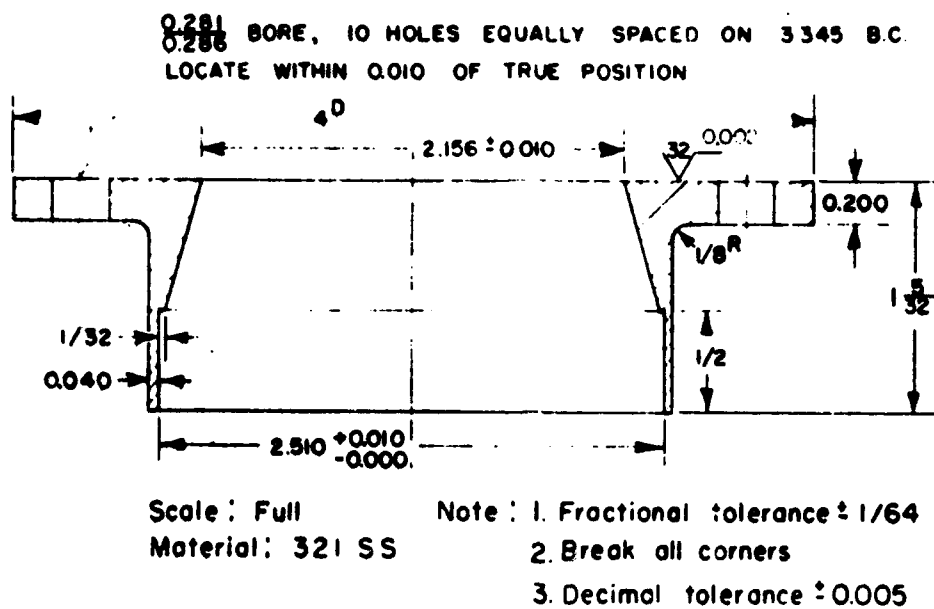
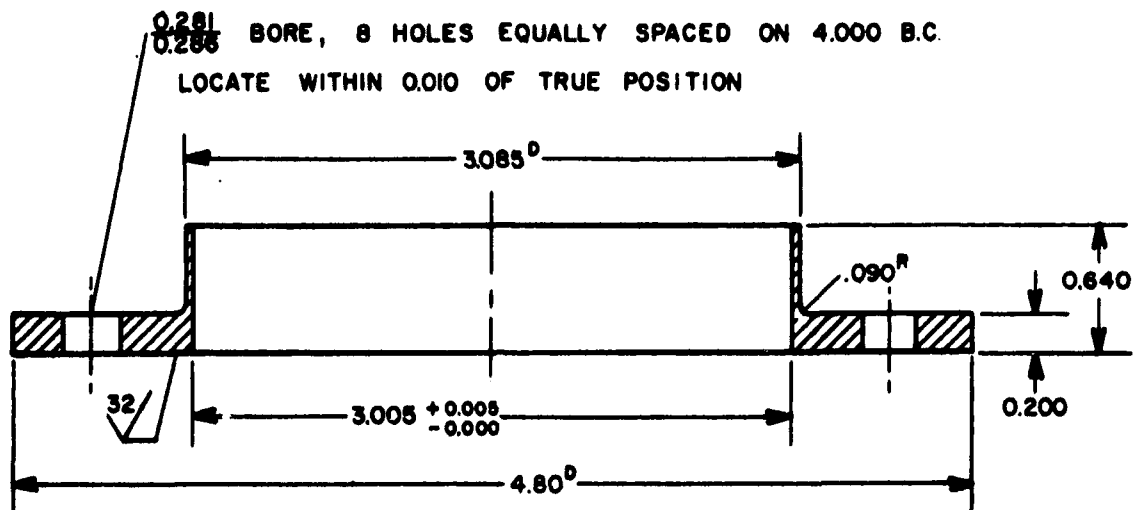


FIGURE 10

Flange for 2.5" O.D. line



Scale: Full

Note: Fractional tolerance  $\pm 1/64$

Material: 304 SS

Decimal tolerance  $\pm .010$ , except  
where noted.

FIGURE 11

**Centaur Flange for  
3" O.D. Line**

TABLE 9. Seal Tests Using Convair Astronautics  
2.5 and 3-inch Flat Flanges

Parco Neoprene O-Rings (Compound 307-50)

<u>Test No.</u>	<u>Flange</u>	<u>O-Ring Size</u>	<u>Torque on Bolts</u>	<u>Comments and Results</u>
5a.	Fig. 11 steel to steel	3.239" I.D. .070" W	75 in-lb	Flange edges just touched. OK warm. Leaked while cooling
5b.	ditto	ditto	180 in-lb	Additional squeeze after flange edges touched. Seal deformed inward to flower shape. Leaked warm.
6.	ditto	ditto	120 in-lb	OK warm. Leaked approx. 1 liter/hr when cold.
39.	Fig. 10 steel to Al.	2.614" I.D. .070" W	125 in-lb	Al. anodized, unpolished. Temp cycled twice, pressure cycled to 160 psig 5 times. <u>No leak at 76°K.</u>
44.	ditto	ditto	ditto	Al. anodized, polished. <u>No leak at 76°K, 160 psig.</u>

(Table 9. Continued on next page)



# Parco Hypalon O-Rings (Compound 921-50)

<u>Test No.</u>	<u>Flange</u>	<u>O-Ring Size</u>	<u>Torque on Bolts</u>	<u>Comments and Results</u>
7.	Fig. 11 steel to steel	3.489" I.D. .070" W.	155 in-lb (Flange edges touch at 50 in-lb)	Pressure cycled 1 atm to 160 psig 40 times during cooldown, 25 times at 76°K. Temp cycled 25 times. <u>No leak.</u>
8.	ditto	3.350" I.D. .070" W	100 in-lb (Flange edges touch at 50 in-lb)	OK warm, held 1 atm at 76°K Small leak at 150 psig, 76°K
41 & 59	Fig. 10 steel to Al	2.614" I.D. .070" W	135 in-lb	Al. not anodized. OK warm. <u>No leak at 76°K, 240 psig:</u> <u>No leak at 20°K, 150 psig.</u>
56a.	Fig. 11 steel to Al	2.864" I.D. .070" W	125 in-lb	Al. not anodized. Used sleeve. OK at 150 psig warm. Leaked during cooldown.
56b.	ditto	ditto	150 in-lb	Same as 56a except torque. <u>No leak warm or 76°K.</u>
60.	ditto	ditto	ditto	Newly machined steel flange. Used sleeve. <u>No leak warm or 76°K, 225 psig.</u>

(Table 9. Continued on next page)

Parco Hypalon O-Rings (Compound 921-50)			
Test No.	Flange	O-Ring Size	Torque on Bolts
37.	Fig. 11 steel to Al	3.239" I.D. .070" W	150 in-lb
			Al. not anodized. Temp cycled 5 times, pressure cycled 10 times. <u>No leak at 76°K, 150 psig.</u>
58.	ditto	ditto	ditto
			Al. not anodized. Used retaining sleeve. <u>No leak at 150 psig and room temp, 76°K, 20°K.</u>
Parco Viton O-Ring (Compound 949-60)			
61.	Fig. 11 steel to Al.	2.864" I.D. .070" W	125 in-lb
			Al. not anodized. Used retaining sleeve. <u>No leak at 150 psig, room temp and 76°K.</u>
62.	ditto	ditto	ditto
Double Mylar with Nichrome Wire Between			
9.	Fig. 11 steel to steel	.0075" Mylar .065" Nichrome 3-1/4" Ring. Diam.	100 in-lb
			15 hot water to LN <sub>2</sub> cycles. 160°F overnight bake. 40 cycles, warm and cold, 150 psig to 1 atm. <u>No leak at 250 psig room temp. No leak at 150 psig 76°K.</u>

(Table 9. Continued on next page)

Double Mylar with Nichrome Wire Between			
Test No.	Flange	O-Ring Size	Torque on Bolts
11.	Fig. 11 steel to steel	.0075" Mylar .065" Nichrome 3-1/4" Ring. Diam.	75 in-lb
			ditto Test 9 except no 160°F bake. No leaks warm or 76°K. 80 pressure cycles.
12a.	ditto	ditto	60 in-lb
			Slight leak both warm and 76°K at 150 psig.
12b.	ditto	ditto	70 in-lb
			No leak warm and 76°K at 150 psig. Leaked cold at 240 psig.
10.	ditto	.005" Mylar .065" Nichrome 3-1/4" Ring Diam.	75 in-lb
			Leaked warm. Mylar cracked when increasing torque.
38.	Fig. 11 steel to steel	.0075" Mylar .050" Nichrome 3-3/16" Ring Diam.	100 in-lb
			OK warm, leaked cold. Warmed and torqued to 125 in-lb, which cut through the Mylar.
40.	Fig. 10 steel to Al	.0075" Mylar .050" Nichrome 2-11/32" Ring Diam.	75 in-lb
			Leaked warm. Torqued to 80 in-lb, which cut through the Mylar.

(Table 9. Continued on next page)

# Miscellaneous Seals

Test No.	Flange	Seal Description	Torque on Bolts	Comments and Results
32.	Fig. 11 steel to steel	R1 Teflon <sup>R1</sup> -coated Pressure actuating Metal core seal	Low. Squeezed to spacer. thick- ness	Seal supplied by Convair. Leak approx. 17 cc/hr at 150 psig, 76°K
46.	Fig. 10 steel to Al	ditto	ditto	Al. anodized and polished. Leak at room temp, 150 psig, was 390 cc/hr.
42.	ditto	ditto	ditto	Al. not anodized. Leak rate at 76°K, 150 psig, was 80 cc/hr.
64.	Fig. 11 steel to Al	R2 Fluorogreen <sup>R2</sup> O-Ring 3" I. D. .070" W.	100, 125, and 150 in-lb	Used sleeve. Req'd 150 in-lb torque to make room temp. seal. Then <u>no leak</u> at 250 psig, 76°K.
66.	ditto	ditto	150 in-lb	Used sleeve. Leaked approx. .04 cc/hr at 250 psig, room temp. Leaked more than .5 cc/hr at 76°K, 175 psig

R 1 du Pont trademark

R 2 Trademark of John L. Dore' Co. for their modified Teflon product.

(1) Seal designated "Rayco", supplied to Convair Astronautics by the Fluorocarbon Co.

The Mylar "sandwich" seal was very reliable when the proper combination of film thickness, compression ring cross section, and compressive force were used. In both types of seal a spacer ring to control the amount of compression would be advantageous. A machined step in one of the flange faces would also serve this purpose. The Mylar seals require only about half as much flange loading as the .070" W elastomers, but this advantage is somewhat offset by the fact that the elastomers can more easily adjust to flange irregularities. Another problem in the use of Mylar film is the possibility of cracking if the compressive stress is too concentrated or too high.

An important factor present in these tests was flexure of the flanges. In every case there was appreciable flexing of the flanges which resulted in a more or less uniform spring loading of the seals. In the case of the extreme lightweight flange, flexing was so non-uniform that a reliable seal could not be achieved. The other flanges flexed into a fairly uniform cone shape (although some of the seals deformed into a flower pattern, showing some bowing between bolts). When the flexing was relatively uniform it served to reduce the initial compression required to maintain the seal during cooldown. Measurements indicated that good seals were being made with 65 to 70% compression of the O-ring, whereas 70 to 80% has been required in previous tests.

An attempt was made to study this spring loading effect with the aid of cone-shaped washers having a known spring constant. The .070" W O-rings of Hypalon compound 921-50 were loaded through 6 of these washers having a spring constant of 61.5 lb/mil. An apparent squeeze of 80% required to hold the seal without washers was reduced to 74% when loaded through the washers. Total force on the 1"

diameter O-ring was 6300 pounds, or 2000 pounds per linear inch of original circumference. Although heavy flanges and bolts were used for this experiment there was still some flexing of the parts, which complicated accurate measurement of O-ring thickness.

The Fluorogreen<sup>[R 1]</sup> seal shown under Miscellaneous Seals in Table 9 holds some promise for applications which cannot use elastomers because of oxygen incompatibility. This modified Teflon<sup>R</sup> material made a good seal in our first test, when used in the same way as elastomer O-rings, even though the plastic was permanently deformed to the shape shown in Figure 9. A second identical test however, was not satisfactory. The leak rate was not excessive, but it built up slowly at 76°K, and was still increasing when it exceeded the scale of the helium leak detector. It is of course possible that the second O-ring was defective, but at present the tests of this material are inconclusive.

### 3.2.5 Recommendations

In summary, it has been shown that elastomers and Mylar film produce satisfactory seals between the heavier Centaur fuel flanges. The possible problems remaining are: 1) vibration testing which was not investigated in the Centaur flanges, and: 2) physical retainment and ease of assembly. Where applicable, based on our present knowledge, it would appear that the present seals problems could best be solved as follows:

[R<sup>1</sup>] Trademark of John L. Dore' Co.

[R] du Pont Trademark

1. Use simple flat flanges with sufficient stiffness to prevent bowing between bolts, but a slight conical flexing of flanges or stretching of bolts (which does not exceed the elastic limit of the materials) is not objectionable and will tend to reduce the required flange loading. Design the flange bolts for a load of about 2000 pounds of force per linear inch of original O-ring circumference. The insides of the bolt holes should not intersect the compressed O-ring but should be kept as close to the O-ring as practical.

2. A normal 32 microinch machined flange surface is suitable. All traces of oil or grease should be removed from the flange faces with solvent.

3. Choose an O-ring (see Table 2 for suggested materials) having a cross section diameter of .070" and a mean ring diameter slightly less than the I. D. of the flanges.

4. Stretch the O-ring around a short thin walled tube (same material as the flanges) which fits snugly inside the flange I. D.

5. Slip this retaining tube inside one flange, with the O-ring resting on the inside shoulder of the flange.

6. Bring the mating flange into position and cross-tighten the bolts until the O-ring is compressed to 75 to 80%.

7. The O-ring will flatten and spread outward at the same time that it is forced tightly against the retaining sleeve. Slight extrusion around the inside corners of the flanges is not objectionable.

8. A .014" thick spacer ring or an equivalent machined step in one of the flange faces will help control the O-ring compression, and will eliminate the need for taking bolt torque readings or O-ring compression measurements.

Current experiments at GDA are investigating various metal and molded elastomer configurations which may offer simple means of physical retainment and field installation.

### 3.3 Ball Bearings for Zero-Gravity Centrifugal Vent Device

All necessary modifications to the ball bearing test apparatus were completed and an experimental program was initiated to verify the reliability of such bearings for the GDA centrifugal vent device. A photograph of the test apparatus is shown in Figure 12. The equipment is shown in position for a bearing test at 20°K. From right to left the main components are a liquid hydrogen supply dewar, a liquid nitrogen precooling heat exchanger, the ball bearing test apparatus and the instrumentation console.

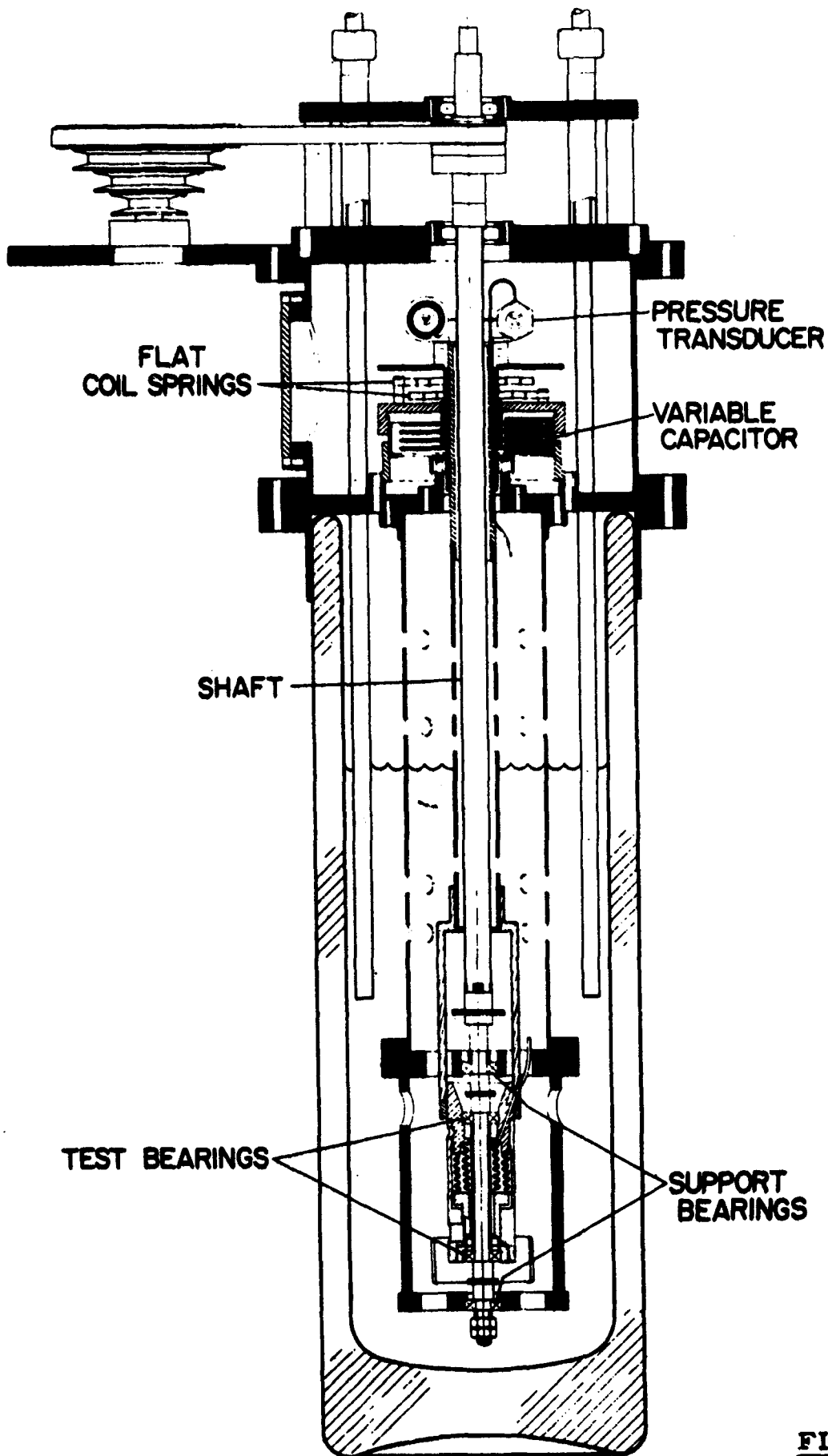
The bearing test apparatus is shown in cross section in Figure 13. A bellows arrangement is provided to apply a thrust load to the test bearings. Torque is monitored by means of a variable capacitor torquemeter. Tests have been made at a constant speed of 9200 rpm. The apparatus shown is primarily used for submerged liquid tests. The major modification for gas tests was to provide gas cooling nozzle rings adjacent to the bearings. These are shown in the exploded view of Figure 14. The outside bearings are support bearings which operate unloaded. The thrust mechanism shown at top center applies the load to the two center test bearings.

The hydrogen gas used to cool the bearing is brought into the tester through an arrangement of heat exchangers and mixing valves to vary the inlet temperature of the gas as required. The coolant gas is piped to the nozzle rings and discharged through 6 holes 0.030 inch diameter at each test bearing. The gas passing thru the holes is operating at subcritical pressure ratios -- normally with a pressure drop of 1 - 2 psi. In the present tests, the entire quantity of gas discharging from the nozzles at the test bearings is assumed to be active in the cooling process.





FIGURE 12  
BALL BEARING TEST APPARATUS



**FIGURE 13**  
**CROSS-SECTION VIEW OF**  
**BALL BEARING TESTER**

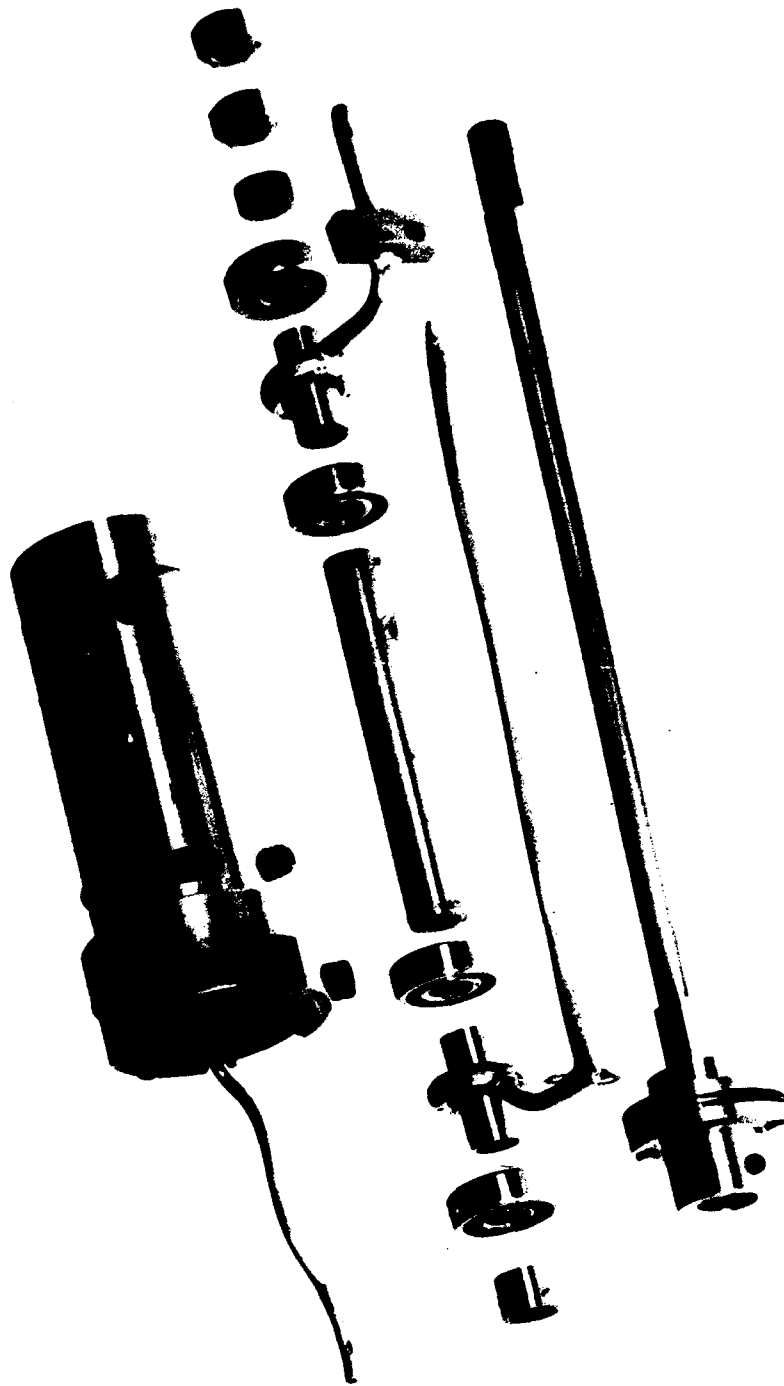


FIGURE 14  
VIEW OF BEARING TEST CHAMBER

The operating requirements on the ball bearings were indicated as follows: speed - 9200 rpm, thrust load - 30 pounds, torque - as low as possible, but not to exceed 5 in. oz. per bearing at operating conditions, life - 20-30 hours minimum. Tests have been conducted to determine if these requirements can be satisfied.

The ball bearings tested have thus far been restricted to 26 mm. O.D. x 10 mm bore bearings having 440 C stainless steel balls and races. The primary separator material was Rulon A (reinforced polytetrafluoroethylene resin which contains a highly inert inorganic silicate base filter). It was selected on the basis of the most successful material determined, thus far, from tests of bearings submerged in liquid nitrogen<sup>1, 2</sup>. Two tests (four bearings) were also performed using carbon-graphite compounds. The latter separators failed by disintegration within 3 hours of initiating the tests. It is difficult to draw conclusions based on these meager experimental data. As time permits, further tests will be made and reported.

The bearings with Rulon-A separators have performed with good success. To date, a total of nearly 200 hours have been accumulated on 10 different bearings. No failures occurred. The criteria for failure has been excessive torque.

- 
1. Wilson, W. A. et al.; Evaluation of Ball Bearing Separator Materials Operating Submerged in Liquid Nitrogen, ASLE Transactions 4, 50-58 (1961)
  2. Brennan, J. A. et al. Testing of Ball Bearings with Five Different Separator Materials at 9200 RPM in Liquid Nitrogen, ASME Paper Number 61-LUBS-18, 8 pages

In the course of the tests it was observed that a definite minimum or critical flow rate of coolant gas was required to prevent excessive torque. When flow rates less than the critical were allowed, the torque increased rapidly. However, the initial torque was recovered if the flow rate was again increased. This behavior is presently being theoretically and experimentally investigated as a function of torque, thrust load and operating temperature in an effort to better understand the operation of ball bearings in a cold, dry gas atmosphere. Modifications have been made to the apparatus to have better control on the applied thrust load.

Ball bearings have been operated with the coolant hydrogen gas at temperatures ranging from 22°K to 310°K. The critical flow rates at specified conditions increased from approximately 1 SCFM (20°C and 1 atm.) (1 SCFM = 0.3 pounds per hour) to 3 SCFM per bearing respectively. Successful operation over the entire temperature range can easily be maintained. The majority of tests have been conducted with hydrogen gas at liquid nitrogen temperature or above. To verify that this temperature is sufficient to be indicative of the problems at lower temperatures, a 21 hour test was made at 22°K. As should be expected, no additional problems were encountered.

In addition to the above 21 hour test, a 55 hour test was made at liquid nitrogen temperature. These constituted "endurance" tests for the present application. A summary of these is indicated below.

Table 10

## Ball Bearing Endurance Tests

## Rulon -A Separators

Temperature of coolant gas - °K	93	22
Total operation - hours	55	21
Coolant gas flow - SCFM/bearing	1.2	1.0
Thrust load - pounds (avg)	45	35
Speed - rpm	9200	9200
Torque - in. oz.	2	1

Bearing wear was measured by the "stick-out" method. The wear in the tests outlined above were within the accuracy of the measuring equipment - 0.0001 inch. One additional quantity was monitored during the course of the endurance tests - starting torque. Since the vent device will be required to stop and restart during flight, it was felt necessary to determine any increase in starting torque. The apparatus was stopped at various intervals and turned both manually and by the drive motor. No appreciable increase from initial values were observed. The starting torque by both methods was approximately 2-3 times the running torque.

Additional endurance tests are not presently planned, but operating time will continue to be accumulated in the course of the bearing parameter study.

#### **4. A Compilation of Thermophysical Properties of Cryogenic Materials**

**R. B. Stewart and V. J. Johnson**

Data sheets containing graphical and tabular presentation of selected values that were in progress at the time of the last report have been substantially completed. These data sheets also include a documentation of the data sources, information on methods of analysis of the data and the basis for selection of the data together with comparisons of any alternate sources. The subjects completed in this group of data sheets were:

1. Compressibility Factor ( $PV/RT$ ) for helium, hydrogen, neon, nitrogen, air and methane.
2. A Temperature-Entropy Chart for neon (temp. range 55-300°K, with pressures to 100 atm. )
3. Liquid-Vapor Equilibrium concentrations of two phase binary systems for helium in hydrogen, nitrogen, methane; hydrogen in nitrogen, carbon monoxide, methane; nitrogen in oxygen, carbon monoxide, argon, and methane.
4. Electrical Resistivity at low temperature of 54 pure metallic elements.
5. Thermal Conductivity Integrals at low temperature of 44 pure metal substances, 36 non-ferrous alloys, 9 ferrous alloys, and 4 glasses and plastics.
6. A Bibliography on the Compressibility Factor, Entropy, and Equilibrium concentrations of two phase binary systems for cryogenic fluids, including: helium, hydrogen, neon, nitrogen, oxygen, carbon monoxide, air, argon, fluorine, methane, as well as a bibliography on the electrical resistance of metals at low temperature. The bibliography is also cross-indexed for these categories.

The above items represent subject matter that was initiated under former sponsorship by Wright Air Development Division of USAF but uncompleted at the time their funding expired.

Additional tasks that were more recently undertaken and are not included in the above groupings of data sheets are:

1. A Temperature-Entropy Diagram for Helium (Temperature range 20° to 300°K, pressure range 0.1 to 100 atm.) (100% completed).
2. The development of an equation of state for helium for the same range of values as well as equations for calculation of thermodynamic properties that are designed to be used in thermodynamic process and system analysis calculations. (100% complete)

(A paper is being prepared for presentation on items 1 and 2 for the Second Symposium on Thermophysical Properties to be held at Princeton University, January 22-26, 1962 and sponsored by the American Society of Mechanical Engineers.)

3. Similar tasks for the preparation of thermodynamic data have also been undertaken for argon and nitrogen.
4. A comprehensive bibliography for the P-V-T, thermodynamic and transport properties of oxygen is nearing completion. This bibliography will contain over 250 citations. (A NBS Technical Memorandum is being prepared for the presentation of this bibliography on the properties of oxygen.)
5. A similar task for bibliographies has also been undertaken for the properties of fluorine.



DEPARTMENT OF COMMERCE

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Electricity. Resistance and Reactance. Electrochemistry. Electrical Instruments. Magnetic Measurements. Dielectrics. High Voltage.

Metrology. Photometry and Colorimetry. Refractometry. Photographic Research. Length. Engineering Metrology. Mass and Scale. Volumetry and Densimetry.

Heat. Temperature Physics. Heat Measurements. Cryogenic Physics. Equation of State. Statistical Physics. Radiation Physics. X-ray. Radioactivity. Radiation Theory. High Energy Radiation. Radiological Equipment. Nucleonic Instrumentation. Neutron Physics.

Analytical and Inorganic Chemistry. Pure Substances. Spectrochemistry. Solution Chemistry. Standard Reference Materials. Applied Analytical Research. Crystal Chemistry.

Mechanics. Sound. Pressure and Vacuum. Fluid Mechanics. Engineering Mechanics. Rheology. Combustion Controls.

Polymers. Macromolecules: Synthesis and Structure. Polymer Chemistry. Polymer Physics. Polymer Characterization. Polymer Evaluation and Testing. Applied Polymer Standards and Research. Dental Research.

Metallurgy. Engineering Metallurgy. Microscopy and Diffraction. Metal Reactions. Metal Physics. Electrolysis and Metal Deposition.

Inorganic Solids. Engineering Ceramics. Glass. Solid State Chemistry. Crystal Growth. Physical Properties. Crystallography.

Building Research. Structural Engineering. Fire Research. Mechanical Systems. Organic Building Materials. Codes and Safety Standards. Heat Transfer. Inorganic Building Materials. Metallic Building Materials.

Applied Mathematics. Numerical Analysis. Computation. Statistical Engineering. Mathematical Physics. Operations Research.

Data Processing Systems. Components and Techniques. Computer Technology. Measurements Automation. Engineering Applications. Systems Analysis.

Atomic Physics. Spectroscopy. Infrared Spectroscopy. Solid State Physics. Electron Physics. Atomic Physics. Instrumentation. Engineering Electronics. Electron Devices. Electronic Instrumentation. Mechanical Instruments. Basic Instrumentation.

Physical Chemistry. Thermochemistry. Surface Chemistry. Organic Chemistry. Molecular Spectroscopy. Molecular Kinetics. Mass Spectrometry.

Office of Weights and Measures.

### BOULDER, COLO.

Cryogenic Engineering Laboratory. Cryogenic Equipment. Cryogenic Processes. Properties of Materials. Cryogenic Technical Services.

#### CENTRAL RADIO PROPAGATION LABORATORY

Ionosphere Research and Propagation. Low Frequency and Very Low Frequency Research. Ionosphere Research. Prediction Services. Sun-Earth Relationships. Field Engineering. Radio Warning Services. Vertical Sounding Research.

Radio Propagation Engineering. Data Reduction Instrumentation. Radio Noise. Tropospheric Measurements. Tropospheric Analysis. Propagation-Terrain Effects. Radio-Meteorology. Lower Atmosphere Physics.

Radio Systems. Applied Electromagnetic Theory. High Frequency and Very High Frequency Research. Modulation Research. Antenna Research. Navigation Systems.

Upper Atmosphere and Space Physics. Upper Atmosphere and Plasma Physics. Ionosphere and Exosphere Scatter. Airglow and Aurora. Ionospheric Radio Astronomy.

#### RADIO STANDARDS LABORATORY

Radio Physics. Radio Broadcast Service. Radio and Microwave Materials. Atomic Frequency and Time-Interval Standards. Millimeter-Wave Research.

Circuit Standards. High Frequency Electrical Standards. Microwave Circuit Standards. Electronic Calibration Center.

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